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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Grain Size in Cast Materials: Delineation, Estimation and Significance

In connexion with plans for standardization of methods of expressing grain size in cast metals and alloys, the Technical Committee of the Institute of British Foundrymen has arranged publication of three relevant reports recently submitted to the Institute by, respectively, the British Non-Ferrous Metals Research Association, the British Steel Castings Research Association and the British Cast Iron Research Association.

A. CIBULA: 'A Note on the Grain Size of Non-Ferrous Castings.'

Brit. Foundryman, 1958, vol. 51, Nov., pp. 581–4.

The influence of grain size and shape of grain, on the properties of wrought non-ferrous alloys, has been studied in detail, but far less information has been published with regard to the effect of grain size in non-ferrous castings, and grain size is rarely specified for such alloys. This note is a summary of information available on this subject, supplementing a report published by the author in *Foundry Trade Journal*, 1952, vol. 93, Dec. 18, pp. 695–703. The main aims of the present report are to consider methods of measuring and reporting grain size which may be suitable as standard procedures for evaluation of cast alloys, and to determine the alloys for which there are preferred grain sizes.

Methods which may be used for revelation of macrostructure in cast non-ferrous alloys are surveyed in a table showing the compositions of etching reagents suitable for use on aluminium-, copper-, lead-, magnesium-, nickel-, tin-, and zinc-base cast alloys. Discussion of methods which may be used for estimation of grain size is followed by a survey of the relatively sparse information available on the influence of grain size on the properties of cast non-ferrous alloys. Difficulties encountered in setting up preferred grain sizes for such materials are considered.

W. J. JACKSON: 'The Measurement and Effect of Grain Size in Steel Castings.'

Ibid., pp. 584–8.

The importance of grain size in relation to the various engineering, physical and mechanical properties of steels has long been recognized: attention is directed to a comprehensive review of the subject published by Ward and Dorn in 1939, and to other relevant literature.

This report contains a critical review of the literature on methods of revealing and assessing austenite grain size, and on the significance of grain size in steel castings. Consideration of the fracture, oxidation, vacuum-evaporation, gradient-quench, martensitic-etch, Zephiran - chloride, bronze - diffusion and McQuaid-Ehn tests for revelation of austenite grain size leads to the conclusion that the martensitic-etch test is to be preferred. A comparison is made of methods of grain-size rating, with some notes on the advantages and limitations of the various techniques which have been proposed. The simplest procedure is a method of rating based on a comparison of the grains in the sample with those covered by standard charts. It is concluded that the significance of austenite grain size in steel castings is not yet fully understood.

H. MORROGH: 'The Problem of Grain Size in Cast Iron.'

Ibid., pp. 588–91.

It is pointed out that, due to the nature of cast irons, the term 'grain size' cannot be applied to these materials in the same sense as that in which it is used in connexion with many steels and non-ferrous alloys. The relevance of the term to the matrix constituents is then discussed, and the influence of ferrite grain size on properties is considered.

The vital significance of form, size and distribution of the graphite phase in cast iron is discussed, and reference is made to the A.S.T.M. graphite-flake size and type chart, which has fulfilled a useful preliminary purpose in defining the microstructure of cast irons. Attention is then directed to recent work which has shown that the structure of the graphite in cast irons can be referred to what is known as a 'eutectic-cell' size, and that by appropriate counting of eutectic cells a good correlation can be obtained between microstructure, mechanical properties, chilling tendency, soundness, etc. It is believed that this concept provides one of the most important methods of quantitatively defining the microstructure of cast iron in relation to its method of manufacture and properties. The fundamental principles of this concept are described and diagrammatically illustrated. The eutectic-cell theory has not yet been fully exploited, and research is currently in progress by the British Cast Iron Research Association. A comprehensive report on the subject is to be published shortly.

Methods for Measurement of Thermal Conductivity of Solids

A. M. ROSS: 'A Literature Survey on the Measurement of Thermal Conductivity of Several Solids, including Uranium Dioxide.'
Atomic Energy of Canada, Ltd., Chalk River Project, Report CRFD-762, Mar. 1958; 39 pp.

In connexion with the requirement for measurement of thermal conductivity of small sintered samples of uranium dioxide at various temperatures, a survey was made of literature relating to the various methods which may be employed for determination of this property. The procedures described in this literature, to which reference is given in a *bibliography* of 142 items, are classified and discussed in sections covering, respectively,

I. Steady-State Methods

Longitudinal heat flow

Parallel-plate methods

Comparative heat flow

Forbes'-bar methods

Radial heat flow

Electrical-heating methods

II. Transient Heat-Flow Methods

The principle of each method is described, and relevant literature is discussed.

As a result of the survey, it is suggested that a comparative heat-flow apparatus be developed for measurements on uranium dioxide in vacuum at temperatures up to 1400°C., and that the comparative heat-flow method now being used in the Chalk River Laboratories be improved in accuracy and further developed for remote-control measurements on irradiated samples. Finally, it is proposed that a transient-state heat-flow comparative method should also be examined.

A helpful general feature of the report is an index giving reference to items of literature (consulted during the search) in which thermal-conductivity values are reported for materials other than uranium dioxide.

The items relevant to thermal-conductivity data on nickel-containing materials are listed below, in order of their appearance in the bibliography:

NICKEL

KING, R. — 'Thermal Diffusivity of Nickel.' *Physical Rev.*, 1915, vol. 6, p. 437.

SCHOFIELD, F. — 'The Thermal and Electrical Conductivities of Some Pure Metals.' *Proc. Royal Soc., Ser. A*, 1925, vol. 107, p. 206.

FRAZIER, R. — 'Further Data on the Thermal Diffusivity of Nickel.' *Physical Rev.*, 1932, vol. 40, p. 592.

VAN DUSEN, M. and SHELTON, S. — 'Apparatus for Measuring the Thermal Conductivity of Metals up to 600°F.' *U.S. Bur. Standards, Jnl. Research*, 1934, vol. 1, p. 429.

STARR, C. — 'An Improved Method for the Determination of Thermal Diffusivity.' *Rev. Scientific Instruments*, 1937, vol. 8, p. 61.

WEEKS, J. and SEIFERT, R. — 'Apparatus for the Measurement of the Thermal Conductivity of Solids.' *Rev. Scientific Instruments*, 1953, vol. 24, p. 1054.

HOGAN, C. and SAWYER, R. — 'The Thermal Conductivity of Metals at High Temperature.' *Jnl. Applied Physics*, 1952, vol. 23, p. 177.

SILVERMAN, L. — 'Thermal-Conductivity Data Presented for Various Metals and Alloys up to 900°C.' *Jnl. Metals*, 1953, vol. 5, p. 631.

POWELL, R. W. — 'Thermal Conductivities of Solid Materials at High Temperatures.' *Research*, 1954, vol. 7, p. 492.

DAVEY, P. and DANIELSON, E. — 'Thermal Conductivity of Pure Nickel.' *I.S.C.* 518, 1954.

MOSS, M. — 'Apparatus for Measuring the Thermal Conductivity of Metals in Vacuum at High Temperatures.' *Rev. Scientific Instruments*, 1955, vol. 26, p. 276.

NICKEL-COPPER ALLOYS

SAGER, G. — 'Investigation of the Thermal Conductivity of the System Copper-Nickel.' *Rensselaer Polytechnic Inst., Engineering and Science Ser.*, No. 27, 1930.

NICKEL-IRON ALLOYS

SILVERMAN, L. — *loc. cit.*

COPPER-NICKEL ALLOYS

SAGER, G. — *loc. cit.*

NICKEL OXIDE

KINGERY, W. *et al.* — 'Thermal Conductivity: X. Data for Several Pure Oxide Materials Corrected to Zero Porosity.' *Jnl. Amer. Ceramic Soc.*, 1954, vol. 37, p. 107.

MONEL

POWELL, R. W. — 'The Thermal and Electrical Conductivity of Metals and Alloys. Part II: Some Heat-Resistant Alloys from 0°-800°C.' *Proc. Physical Soc. (Lond.)*, 1936, vol. 48, p. 381.

SILVERMAN, L. — *loc. cit.*

STAINLESS STEELS

HOGAN, C. and SAWYER, R. — *loc. cit.*

SILVERMAN, L. — *loc. cit.*

PRZYBYCIEN, W. and LINDE, D. — 'Thermal Conductivities of Gases, Metals and Liquid Metals.' *U.S. Atomic Energy Commission, Knolls Atomic Power Laboratory, Report M-WMP-1*, 1957.

DEISSLER, R. and EIAN, C. — 'Effective Thermal Conductivity of Magnesia, Stainless Steel and UO_2 Powders in Various Gases.' *Nat. Advisory Committee for Aeronautics, Research Memorandum E-53CO3*, 1953.

Storage Batteries: Characteristics and Applications

See abstract on p. 6.

NICKEL

Revised A.S.T.M. Specifications for Nickel and Nickel Alloys

The 1958 edition of *A.S.T.M. Standards, Part II, Non-Ferrous Metals*, includes revised specifications for a number of nickel-containing materials, amplifying and/or modifying earlier schedules. Eight of these specifications have been reprinted by INTERNATIONAL NICKEL COMPANY, INC, and are correlated with the schedules and designations used within the INCO organization to cover the respective materials. The metals and alloys to which the schedules relate are listed below, with brief notes on the scope of the specifications.

'Nickel-Copper Alloy Plate, Sheet and Strip.'
A.S.T.M. Designation B 127-58*T* (*INCO A*509*A*).

Covering sheet and strip of material conforming to the limits: nickel 63·0–70·0, iron 2·5 max., manganese 1·25 max., carbon 0·3 max., silicon 0·5 max., sulphur 0·024 max., per cent., copper remainder. The alloy 'Monel' falls within this composition range.

'Nickel Rod and Bar.'
A.S.T.M. Designation B 160-58*T* (*INCO A*500*A*).

Covering 'A' nickel and low-carbon nickel.

Compositional percentage limits for both materials are: nickel 99·0 min., copper 0·25 max., iron 0·40 max., manganese 0·35 max., silicon 0·35 max., sulphur 0·01 max. For the 'A' grade the carbon is 0·15 max., for the low-carbon grade the limit is 0·02 max. The specification covers nickel in the form of rounds, squares, hexagons and rectangles.

'Nickel Seamless Pipe and Tube.'
A.S.T.M. Designation B 161-58*T* (*INCO A*501*A*).

Covering 'A' nickel and low-carbon-nickel cold-drawn seamless pipe and tube. Composition requirements as in *B*160–58*T*; *see above*.

'Nickel Plate, Sheet and Strip.'
A.S.T.M. Designation B 162-58*T* (*INCO A*502*A*).

Covering 'A' nickel and low-carbon nickel.

Composition requirements as in *B* 160–58*T*: *see above*.

'Seamless Nickel and Nickel-Alloy Condenser and Heat-Exchange Tubes.'
A.S.T.M. Designation B 163-58*T* (*INCO A*503*A*).

Covering condenser tubing in nickel and low-carbon nickel, 'Monel' (nickel-copper alloy), 'Inconel' (nickel-chromium-iron alloy).

Compositional limits are as follows:

For the nickel tubes: 'A' nickel and low-carbon nickel (*see B* 160–58*T above*)

„ „ 'Monel' „ : as in *B* 127–58*T* (*see above*)

„ „ 'Inconel' „ : nickel 72·0 min., copper 0·5 max., iron 6·0–10·0 manganese 1 max., carbon 0·15 max., silicon 0·5 max., sulphur 0·015 max., chromium 14·0–17·0, per cent.

'Nickel-Copper Alloy Rod and Bar.'
A.S.T.M. Designation B 164-58*T* (*INCO A*504*A*).

Covering two grades of alloy, 'Monel' and 'R Monel'.

Compositional limits: nickel 63·0–70·0, iron 2·5 max., manganese 2·0 max., carbon 0·3 max., silicon 0·5 max. For 'Monel' the sulphur limit is 0·024 max.; for 'R Monel' (the free-machining grade) the range is 0·025–0·60 per cent. In both cases the remainder is copper.

'Nickel-Copper Alloy Seamless Pipe and Tube.'
A.S.T.M. Designation B 165-58*T* (*INCO A*505*A*).

Covering pipe and tube of 'Monel'.

Compositional limits as for 'Monel' in *B* 164–58*T*: *see above*.

'Nickel-Chromium-Iron Alloy Rod and Bar.'
A.S.T.M. Designation B 166-58*T* (*INCO A*506*A*).

Covering material conforming to the 'Inconel' composition: *see B* 163–58*T above*.

'Nickel-Chromium-Iron Alloy Seamless Pipe and Tube.'
A.S.T.M. Designation B 167-58*T* (*INCO A*507*A*).

Covering pipe and tube of 'Inconel'.

Compositional limits as in *B* 163–58*T*: *see above*.

'Nickel-Chromium-Iron Alloy Plate, Sheet and Strip.'
A.S.T.M. Designation B 168-58*T* (*INCO A*508*A*).

Covering plate, sheet and strip in 'Inconel'.

Compositional limits as in *B* 163–58*T*: *see above*.

Elastic Modulus of Nickel-Copper Alloys

V. A. PAVLOV, N. F. KRIUTCHKOV and I. D. FEDOTOV: 'Relationship of Temperature to Elastic Modulus in Nickel-Copper Alloys.'

Physics of Metals and Metallography, 1957, vol. 5, No. 2, pp. 160–1.

Curves show modulus/temperature relationships over the range 77° to 900°K. (−196° to 627°C.) for nickel, and 77° to 1000°K. (−196° to 727°C.) for nickel-copper alloys containing, respectively, 10, 20 and 40 per cent. of copper. The curve for nickel is in close agreement with the results reported by KÖSTER (*Zeitsch. f. Metallkunde*, 1948, vol. 39, p. 1). Elastic modulus of the alloys steadily decreases with increasing percentages of copper in solid solution.

The Nickel/Nickel-Chloride System

J. W. JOHNSON, D. CUBICCIOTTI and C. M. KELLEY: 'Interactions of Metals with their Molten Salts. I. The Nickel/Nickel-Chloride System.'

Jnl. Physical Chemistry, 1958, vol. 62, Sept., pp. 1107–9.

Nickel metal has been found to have appreciable solubility in molten nickel chloride. The depression of the freezing point of nickel chloride (1009·1°C.) by the dissolved metal was measured: a eutectic point was found at 977·5°C. and 9 mole per cent. nickel. The constitution of the solution is discussed in the light of the depression of the freezing point.

Interdiffusion of Aluminium and Nickel

L. S. CASTLEMAN and L. L. SEIGLE: 'Layer Growth during Interdiffusion in the Aluminium-Nickel Alloy System.'

Trans. Metallurgical Soc., Amer. Inst. Mining and Metallurgical Engineers, 1958, vol. 212, Oct., pp. 589–96.

Formation and growth of intermetallic layers, which is of considerable importance in connexion with diffusion-bonding and cementation processes, has been extensively studied on a qualitative basis, but few quantitative data are available. A survey of conclusions to date, with a report of experimental work, published by LUSTMAN and MEHL in 1942 (*Trans. Amer. Inst. Mining and Metallurgical Engineers*, vol. 147, pp. 365–95) is used as an introduction to the present paper.

The development of reactor technology has brought renewed interest to bear on diffusion-bonding problems as occurring in the manufacture of clad fuel elements and other structural components of reactors. The literature pertinent to this aspect of the subject is considered by the authors, with special reference to the significant finding, reported by Storchheim *et al.*, that pressure exerts a marked effect on bond strength and upon the thickness of the intermediate alloy layers found at the aluminium-nickel interface. In the aluminium-nickel system increasing pressure decreases the thickness of the layers and improves bond strength, and extension of the investigations to other systems containing intermetallic phases has shown that a similar pressure effect exists, for example, in the uranium-nickel system. In view of the important implications, both practical and theoretical, of this pressure effect, research was initiated to investigate the phenomenon in some detail. The first phase of the work was concerned with the kinetics of growth of the intermediate alloy phases in the aluminium-nickel system. Specifically, the increases in thickness of the intermetallic layers were determined as functions of interdiffusion time, interdiffusion temperature, and applied pressure in aluminium-nickel diffusion couples. In addition, studies of growth kinetics of layers were made in couples in which interdiffusion occurred between aluminium or nickel and the $\gamma(Ni_2Al_3)$ intermediate alloy phase which exists in the aluminium-nickel alloy system. The results obtained are reported in this paper.

The materials from which the disc diffusion couples were prepared were high-purity (99·99%) aluminium and vacuum-melted electrolytic nickel. Thickness of the discs ranged from 0·015 to 0·050-in. (0·375–1·25 mm.). The coupled discs were annealed in the temperature range 400°–625°C., for times up to 340 hours and under pressures up to 5 t.s.i. (8 kg./mm.²). Most of the experiments carried out at atmospheric pressure were made on couples prepared by spot welding. Those used in the elevated-pressure experiments were made in a hot-pressing apparatus, illustrated details of which are given in the paper.

Conclusions

'The results demonstrate that all phases which are thermodynamically stable at the diffusion temperature appear in the interdiffusion zone at 600°C.: these phases are the $NiAl_3$ (β), Ni_2Al_3 (γ), NiAl (δ), and Ni_3Al (ε) intermetallic compounds. The rate-controlling factor affecting the kinetics of growth of the γ phase in the early stages is not yet clear; after a minimum thickness of 0·025 to 0·030 mm. is reached, however, volume diffusion controls layer growth. It is estimated that the chemical diffusion coefficient is $9{\cdot}1 \times 10^{-10}$ sq. cm. per sec. and that the heat of activation for diffusion is 31,000 cal. per mol. An applied pressure of 5 t.s.i. (8 kg./mm.²) reduces the diffusion rate by 27 per cent. Volume diffusion controls the growth kinetics of the β phase after a minimum thickness of 0·040 mm. is reached; it is estimated that the 600°C. diffusion coefficient is about $1{\cdot}8 \times 10^{-11}$ sq. cm. per sec. and that the heat of activation is approximately 27,000 cal. per mol. Tentatively, it appears that pressure affects growth of the β phase much less than the γ phase. The growth kinetics of the δ and ε phases also appear to be volume diffusion controlled.'

Local-Cell Action during Scaling of Nickel

C. ILSCHNER-GENSCH: 'Local-Cell Action during the Scaling of Metals, II.'

Jnl. Electrochemical Soc., 1958, vol. 105, Nov., pp. 635–8.

It is known that oxidation of nickel proceeds according to the overall reaction

$$Ni(s)+\tfrac{1}{2}O_2(g)=NiO(s).$$

Where, as in this case, a dense oxide film is formed oxidation can advance only by diffusion of the reactants across the oxide film to each other. It has been suggested by Wagner that the migrating particles are ions and electrons rather than atoms. In the nickel oxide electronic conductivity prevails and the rate of oxidation is therefore determined essentially by the ionic conductivity of the oxide. The rate constant for oxidation of nickel, at 780°C. and 0·1 atm. O_2 partial pressure, has been determined by various authors to be $k=10^{-12}$ to 10^{-11} g^2 $cm.^{-4}$ $sec.^{-1}$ for specimens of various degrees of purity.

This paper is concerned with reactions occurring when nickel is covered by a borate melt, in which conditions oxidation can take place only inasmuch as oxygen dissolves in the borate melt and diffuses from the outer surface of the melt to the metal/salt interface. The relatively low solubility of oxygen molecules in a borate melt results in a low transport rate and, therefore, in a low rate of oxidation. It is shown, however, that if the nickel sample is in electrical contact with an electronic conductor, e.g., a platinum or other noble-metal gauze, which extends up to the metal/oxygen interface, nickel is attacked rapidly, by virtue of local-cell action. In this case electron transfer is accomplished by the metal gauze, and ions migrate through the borate melt. Electrochemical measurements show that the reaction is controlled mainly by polarization of the cathode, where oxygen molecules are reduced to ions.

The exact mechanism of the observed phenomena is not yet fully understood, but some suggestions are made for a hypothesis which is subject to experimental confirmation or modification. It is pointed out that the observations made and the deductions to be drawn from them are of interest in relation to the 'catastrophic oxidation' liable to occur in alloys on which the scale consists of one or several solid oxides and a liquid phase. In such cases ionic conduction may take place in the liquid phase, and electronic conduction in the solid oxides. The rate may be controlled by the cathodic reduction of molecular oxygen (as in the experiments reported in this paper) and may, it is believed, be even greater than those observed in this work because the solid oxides may form a sponge involving numerous pores filled with liquid phases, whereby a large cathodic area is provided. It is considered that local-cell action may account for relatively high rates of oxidation even without the occurrence of different valence states of elements such as iron or vanadium, but the presence of such elements may lead to an even higher rate of oxidation.

'Thermal Regeneration' of Nickel after Low-Temperature Oxidation

R. M. DELL: 'The 'Thermal Regeneration' of Nickel, Copper and Cobalt after Low-Temperature Oxidation.'

Jnl. Physical Chemistry, 1958, vol. 62, Sept., pp. 1139–41.

It has been known since the work of Russell and Bacon (1932) that when a reduced nickel or copper powder is saturated with oxygen at 0°C. and then heated *in vacuo* at a suitably high temperature (200°–400°C.) it is capable subsequently of adsorbing a further quantity of oxygen at 0°C., even though no gas was desorbed during the thermal treatment. This effect, termed 'thermal regeneration', has recently been the subject of investigation by Zettlemoyer *et al.*, who studied the phenomenon on powders of cobalt, copper and nickel (see abstracts in *Nickel Bulletin*, 1958, vol. 31, Nos. 2 and 3, pp. 40 and 69).

The present author summarizes the mechanism of the adsorption phenomena suggested by Zettlemoyer and his co-workers, but points out that alternative mechanisms may be envisaged with regard to thermal regeneration of a metal surface covered by a *thin* oxide film formed at room temperature or below. The experimental work recorded (done on the same three metals) leads to the conclusion that recrystallization of the thin oxide film formed at low temperatures plays a significant rôle in thermal regeneration. It is possible that this factor is more important for nickel than for copper or cobalt. The nature and extent of the recrystallization will depend on the metal concerned and on the severity of the thermal treatment undergone.

Sintering of High-Purity Nickel Oxide

Y. IIDA: 'Sintering of High-Purity Nickel Oxide.'

Jnl. Amer. Ceramic Soc., 1958, vol. 41, Oct., pp. 397–406.

It is known that some metal oxides, when heat-treated, show a departure from stoichiometric composition during oxidation or reduction, resulting in an excess of one or other constituent in the crystal lattice. It is reported that the rate of sintering may be associated with the lattice defects. The aim of the work described was to establish the relationship between densification and grain growth of high-purity nickel-oxide powders (in the form of compacted bars) during sintering, and to clarify the effects, on rate of sintering, of variation in the atmosphere used.

The scope of the investigation is outlined below.

(1) Determination of the influence of time and temperature of sintering on shrinkage of the compact. Data are reported for sintering temperatures up to 1600°C. To establish the effects of time of sintering, isotherms of shrinkage of NiO were obtained over the range 1000°–1400°C.

(2) Investigation of the relationship between grain growth and densification.

(3) Calculation of the activation energy of grain growth.

(4) Investigation of the influence, on densification and on grain growth, of sintering *in vacuo*, in argon, in oxygen, and in air. A comparative study was made of densification of ZnO powders sintered *in vacuo* and in argon.

The effects of variation in sintering temperature, compacting pressure, and atmosphere are well illustrated by micrographs.

From the results of the investigation the author draws the following conclusions:

'(1) The sintering of NiO powders may be divided qualitatively into three stages. In the first stage, densification and grain growth do not occur appreciably; in the second stage, densification is rapid but grain growth is slow; in the third stage, there is no appreciable change in density, and grain growth is rapid.

'(2) Grain growth during the third stage follows the empirical equation $D^2=Kt$, where D is the grain diameter, K is the rate constant, and t is the sintering time. The activation energy of grain growth is 55 Kcal. per mole.

'(3) The shrinkage and weight loss during sintering *in vacuo* exceed those in argon, air, and oxygen.

'(4) The grains of compacts sintered in air are the largest and are round. The grains of compacts sintered in argon and in oxygen are identical in size and are triangular in shape. Compacts sintered *in vacuo* have no grains.

'(5) The densification of ZnO compacts during sintering *in vacuo* is also greater than that in argon.

'(6) The lattice defect leading to semiconductor properties is not related to densification and grain growth during the sintering of NiO compacts.'

Compatibility of Nickel with Graphite at Elevated Temperatures

See abstract on p. 20.

Storage Batteries: Characteristics and Applications

C. K. MOREHOUSE, R. GLICKSMAN and G. S. LOZIER: 'Batteries.'

Proc. Inst. Radio Engineers, 1958, vol. 46, Aug., pp. 1462–83.

This paper contains a comprehensive historical and technical review of battery development, dealing in detail with the structural characteristics, performance capacity and uses of all classes of battery. The survey covers not only batteries commercially available but also important types now under development. Various nuclear and solar batteries are also included, giving a comparison of such units with the conventional electrochemical types. The section relating to secondary batteries contains an illustrated critical review of the nature, advantages and limitations of nickel/iron and nickel/cadmium alkaline batteries.

The review is based on a wide survey of the literature to which reference is given in a *bibliography* of 108 items.

Determination of Nickel by Solvent Extraction

P. D. BLUNDY and M. P. SAMPSON: 'The Determination of Nickel by a Solvent-Extraction Method.'

Analyst, 1958, vol. 83, Oct., pp. 558–61.

The method described provides for the rapid determination of nickel in solutions containing uranium, thorium, copper, iron and chromium. The aqueous insoluble 4-methyl*cyclo*hexane-1:2-dionedioxime complex of nickel is extracted with toluene, and the optical density of this phase is measured with a Spekker absorptiometer, using a Hilger H.556 filter. Interference by copper is suppressed with thioglycollic acid, and tartaric acid is used to prevent formation of iron thioglycollate. Tartrate also prevents the hydrolysis of thorium.

Colorimetric Determination of Nickel

H. B. JONASSEN, V. C. CHAMBLIN and V. L. WAGNER: 'Disodium Ethyl Bis(5-tetrazolylazo)acetate Trihydrate Colorimetric Reagent for Nickel and Copper.'

Analytical Chemistry, 1958, vol. 30, Oct., pp. 1660–3.

During investigation of the co-ordinating tendencies of disodium ethyl bis(5-tetrazolylazo)acetate trihydrate it was observed that it forms a water-soluble, highly coloured complex with the nickel (II) ion. This reaction has been made the basis for quantitative colorimetric determination of nickel in the presence of cobalt and iron. The compound also forms a stable, highly coloured 1:1 complex with the copper (II) ion: this complex has a high absorbance value, even at low concentrations. Of the common cations of the first transition-metal series, only cobalt (II) interferes. Means for elimination of this interference are included in the details of analytical procedure given in this paper.

Gravimetric Separation of Copper, Nickel and Zinc

G. C. KRIJN: 'Quantitative Gravimetric Separation of Copper, Zinc and Nickel.'

Chemisch Weekblad, 1958, vol. 54, Sept. 20, pp. 499–500.

The author describes a method in which copper is precipitated as sulphide in acid solution with thioacetamide and, after the addition of 'Complexone' to the ammoniacal solution of zinc and nickel, zinc is liberated by adding a calcium salt, while nickel is kept in solution.

ELECTRODEPOSITION AND OTHER COATING METHODS

Black-Nickel Plating

'A New Black-Nickel Plating Process.'
Metal Finishing Jnl., 1958, vol. 4, Nov., pp. 436–7.

Black-nickel coatings produced from conventional solutions are subject to certain limitations, e.g., deposits of the thickness required to ensure satisfactory abrasion-resistance tend to be of low ductility and to show relatively poor adhesion. A further disadvantage is that the composition of the solution and the operating conditions are critical if uniform coatings of full black colour are to be produced.

Extensive research carried out by the International Nickel Company, Inc., with a view to eliminating these undesirable features, has culminated in development of an improved process which is covered by *U.S. Pat.* 2,844,530 and is the subject of corresponding patents and applications in all the main industrial countries. In the course of study of many modifications of solution and plating conditions, it has been found that use of chlorides (in place of the sulphates generally used in black-nickel plating) is an important feature. Particulars of the preferred solutions and of working conditions are given below:

	g./L.
Nickel chloride ($NiCl_2.6H_20$)	75
Ammonium chloride (NH_4Cl)	30
Sodium thiocyanate (NaCNS)	15
Zinc Chloride ($ZnCl_2$)	30

pH	5·0
Temperature	Room
Current density	1·5 amp./sq. ft. (0·16 amp./dm.²)

In contrast to the critical proportioning of the constituents in sulphate solutions, it has been found that good black coatings are consistently obtained irrespective of the concentration of ammonium chloride, sodium thiocyanate and zinc chloride, within the ranges indicated. The nickel-chloride content is, however, more critical and it was found that high concentrations (100 to 150 g./L.) require higher current densities (3·0–6·0 amp./sq. ft.; 0·32–0·65 amp./dm.²) to achieve dense black coatings. This limitation is by no means always disadvantageous, since at low current density a minimum plating time of 15 minutes is required to obtain a black coating; shorter times give films so thin that interference colours are seen. Higher current densities will give black deposits in proportionately shorter times.

Changes in pH within the range 2·3 to 5·5 do not affect the colour of the deposit.

The dense black coatings obtained may, if desired, be made more attractive by a light buffing with grease. Etching will produce a matte jet-black coating such as would be suitable for applications in which light reflection must be at a minimum.

Tests reported in this note show that the deposits from the chloride solutions have better abrasion-resistance, adhesion and ductility than deposits from the conventional sulphate solution. Adhesion is enhanced by pre-treating the basis metal (either wrought or electrodeposited nickel) in the acid all-chloride nickel strike bath, similar to that used to obtain adherent deposits on stainless steel. The most effective pre-plating treatment for optimum adhesion is cathodic alkaline cleaning, followed by the acid all-chloride nickel (anodic/cathodic) strike. Such a strike treatment is recommended also prior to black-nickel plating of stainless steels.

On exposure to contaminated atmospheres, black-nickel coatings, whether deposited from sulphate baths or from improved chloride solutions, suffer loss of colour and deteriorate to a mottled grey. A top coating of clear lacquer has been found to give adequate protection, and it is recommended that such protection be given for all black-plated components except those used in very mild indoor atmospheres.

Development of Improved Barrel-Nickel-Plating Techniques

B. E. BUNCE: 'Some Developments in Barrel Nickel Plating.'
Electroplating and Metal Finishing, 1958, vol. 11, Nov., pp. 389–97.

Opinions recorded in the literature are at variance with respect to the extent to which electrolytes used in vat nickel plating may be adapted for use in barrel plating. Stirling, for example, suggests that any solution that has proved satisfactory for vat plating is also suitable for barrel plating, provided that rather higher concentrations are used. At the other extreme, Wallbank is of the opinion that such solutions are not adaptable to barrel plating, on account of the wide variation in operating conditions. Electrolytes suggested for barrel plating have, however been based, in general, on the use of more highly concentrated versions of conventional plating solutions. The present paper opens with a critical review of the literature available on barrel nickel plating, divided into sections on Watts-type solutions, bright-nickel-plating solutions, high-chloride and high-sulphate solutions, and equipment and procedure employed when using the respective electrolytes.

These solutions and processes were developed mainly on an empirical basis. The author's own investigations in this field were made in an attempt to apply a more systematic experimental approach to development of improved barrel-nickel-plating processes.

The investigations were carried out in two phases. The first part of the work, which related to plating using barrels of semi-submerged-horizontal and

oblique types, centred on solutions of a nickel-sulphate/nickel-chloride/boric-acid basis composition. Addition of saccharin improved the brightness of the coatings deposited from this solution, but lowered the conductivity. Introduction of magnesium sulphate was found to improve the conductivity of the solutions: under similar operating conditions the total current was raised from 45–50 amp. to 50–55 amp. Operating the solution at higher temperatures and increasing the size of the holes in the barrel enabled the current to be raised still further. A solution of the following composition gave excellent bright-nickel deposits: $NiSO_4.7H_2O$ 250, NaCl 20, Na_2SO_4 35, H_3BO_3 25, saccharin 3, g./L. Operated at a temperature of 100°F. (38°C.) and a pH of 5·7, a current of 90–100 amps could be obtained at 10 volts with a plating load of approximately 60 sq. ft. (5·5 m.²). A two-hour plating cycle gave an average deposit thickness of 0·0001 in. (0·0025 mm.). This electrolyte, which was one of those investigated in the second phase of the programme, for use in fully-submerged barrel nickel plating, proved unsuitable for use with current densities higher than 2 amp./sq. ft. (0·2 amp./dm.²). Other experiments with modified bright-nickel-plating solutions showed that the success of their application to barrel plating depended to a large extent on the resistance across the barrel panels, i.e., the greater the area of holes, the better the deposits and the higher the current density obtained. For this reason, a pre-fabricated 'Perspex' barrel with particularly thin panels and a large hole area was substituted for the ebonite moulded barrel previously used. The solution finally evolved contained (g./L.) $NiSO_4.7H_2O$ 300, $NiCl_2.6H_2O$ 50, H_3BO_3 40. The brightener consisted of an amino beta-naphthalene sulphonic-acid derivative with small quantities of cadmium and cobalt sulphates. The operating conditions were: temperature 130°F. (54°C.), pH 4·4, current density 3–4 amp./sq. ft. (0·32–0·43 amp./dm.²), potential difference 12 volts.

Occurrence of laminated deposits, believed to be due mainly to the intermittent make and break of the electrical contacts, was minimized by sheathing the standard rubber-covered flexible contact cables with P.V.C. tubing. 'Perspex' barrel-lid clamps with removable clamping blocks were devised to overcome the breakage of, and attack of the plating solution on, the normal rubber- or polythene-covered steel clamps.

In conclusion, the author mentions two proprietary barrel-plating solutions, 'Nibrite' and 'Efco-Udylite 4 B1H', which produce excellent nickel deposits and which are now in exclusive use.

Stress in Electrodeposited Nickel

T. P. HOAR and D. J. ARROWSMITH: 'Stress in Nickel Electrodeposits.'

Trans. Inst. Metal Finishing, 1958, vol. 36, Autumn, pp. 1–6.

The paper was abstracted in preprint form in *Nickel Bulletin*, 1958, vol. 31, No. 6, p. 162.

Oil Pores in Electrodeposited Nickel Coatings

K. SOMMER: 'Some Observations on the Formation of Oil Pores in Nickel Electrodeposits.'

Metal Finishing Jnl., 1958, vol. 4, Nov., pp. 438–40.

The subject matter is closely similar to that of the article published by the author in *Metall*, 1957, vol. 11, pp. 166–7: see *Nickel Bulletin*, 1958, vol. 31, No. 1, p. 5.

Bright-Nickel Plating of Typewriter Typebars

L. ADÈS: 'Bright-Nickel Plating Typewriter Typebars.'

Electroplating and Metal Finishing, 1958, vol. 11, Nov., p. 409.

This note gives the composition of the bright-nickel solution, and full details of the operational conditions and the plating sequence, used by the Société Continentale Parker to nickel-plate typebars of typewriters. The bar itself is of mild steel, which is cyanide-hardened, and finally plated in a conventional dull-nickel solution, to prevent oxidation. The other component, the letter, also of mild steel, is barrel-plated and then soldered to the bar. The composite has then to be coated with a second nickel deposit. No difficulty was encountered when this second coating was deposited from a dull-nickel-plating solution. However, when a change was made to bright-nickel plating for this final step, the stringent requirements with respect to thickness of deposit (5 microns at the edges, 2 microns elsewhere), and the necessity of obtaining adequate adhesion between the two deposits, gave rise to difficulties. Deposits produced from a solution of the composition listed below, operated under the conditions indicated, have fully satisfied all requirements.

	g./L.
Nickel sulphate	20
Sodium sulphate	40
Magnesium sulphate	30
Sodium chloride	10
Boric acid	20
'Udylite Bright-Nickel Addition No. 41'	10 ml./L.
'Udylite Bright-Nickel Addition No. 2RL'	½ ml./L.
Special Addition Agent	10 ml./L.

pH	5·5
Temperature	30–40°C.
Current density	about 1 amp./dm.²

Plating for ten minutes produces a deposit of the required thickness.

Plating of Automobile Hub Caps

'Automatic Polishing, Washing and Plating of Hub Caps.'

Metal Finishing Jnl., 1958, vol. 4, Oct., pp. 381–5, 388.

The article describes plant recently installed by the Ford Motor Company, Ltd., at their Basildon works,

for polishing, pressure washing and bright copper-, nickel-, and chromium-plating of motor-car hub caps. The plant is designed to process 240 caps per hour, under the control of one operator.

The cycle involved comprises cleaning, cyanide-copper plating, followed by plating from a copper-sulphate bath, bright-nickel plating in a 'Super Gleamax' solution, and final chromium plating from a 'Zonax' bright-plating solution.

As part of the Company's policy of rigid control of quality, a proportion of the plated caps are checked for thickness of deposit, using micrometer measurements: some typical thicknesses are quoted in the paper. Test hub caps are also subjected to a minimum of 100 hours' salt-spray test.

The article is illustrated by many photographs.

Tin-Nickel Plating of Watch Parts and Instruments

'Tin-Nickel on Watches and Drawing Instruments.'
Tin and Its Uses, 1958, Autumn, pp. 1-3.

Tin-nickel alloy plating, the properties of which have been described in various technical papers during the past ten years, is now becoming firmly established as a surface finish in applications for which its tarnish- and corrosion-resistance, hardness, abrasion-resistance, low coefficient of friction and other characteristics render it peculiarly suitable.

Among the successful applications of this type of plating is its use (matte or bright) on watch parts: here the throwing power of the plating solution and the corrosion-resistance of the coating are particularly advantageous. The tin-nickel plating resists the corrosive and staining effect of moisture from the fingers of the watchmakers and, throughout the life of the watch, prevents finger marking and corrosion, for example, by condensation. The solution 'throws' successfully between closely set gear teeth, and into small pinion holes, giving an even coating, the anti-frictional qualities of which are of considerable significance. In addition, the alloy coating has the property of retaining films of lubricating oil without being corroded by them even after prolonged contact. Full advantage is thus gained from even a trace of oil, and the plated watch parts will work without deterioration for a very long period.

It is noted that an ordinary wrist watch may include 50-100 pinions, cog wheels, pins, screws, levers, barrels, springs, pawls and ratchet wheels. The thickness of coating plated onto these very fine parts must be strictly controlled: the thickness ranges up to 0·0003 in. (0·0075 mm.) for the larger components, whilst for the smallest screws 0·00003 in. (0·00075 mm.) is in many cases adequate. The possibility of depositing tin-nickel alloy coatings on a variety of basis metals is an advantage in obtaining a uniform surface of good colour in the finished mechanism, and it is a useful feature that the colour of the plating tones well with stainless steel.

It is recorded that three well-known watchmaking firms (situated respectively, in Switzerland, U.S.A. and Scotland) are now regularly using this type of finish, with entirely satisfactory results. This article includes illustrations of typical plated parts.

Tin-nickel coatings have also been adopted by a number of firms making precision instruments such as telescopes, drawing instruments and microscopes, in all of which resistance to finger-marking is an outstanding advantage. One producer of drawing instruments reports that the standard thickness of plating used is 0·0003 in. (0·0075 mm.): the alloy finish has been registered by that firm under the name 'Lansin'.

Polishing and Plating of Zinc-base Die Castings

W. H. SAFRANEK and C. L. FAUST: 'Improved Electroplated Finishes for Zinc Die Castings.'
Plating, 1958, vol. 45, Oct., pp. 1027-37.

This paper presents an account of progress in two phases of a research being conducted at Battelle Memorial Institute, on behalf of the American Zinc Institute:

(1) Study of electropolishing and electroplating procedures by which the costs involved in finishing die castings may be reduced.

(2) Study of plating procedures by which the corrosion-resistance of electroplated diecastings may be improved.

Detailed investigation of surface finish obtainable by electropolishing of (1) mechanically polished and (2) unpolished die castings proved the suitability of an alkaline polishing solution, but, although electropolishing had a useful smoothing effect, none of the die castings was fully bright after plating with copper (0·0003 in.; 0·0075 mm.) + nickel (0·0005-0·0008 in.; 0·0125-0·02 mm.). The need therefore arose for development of an electroplating technique which would give good levelling, thus supplementing the smoothing obtained by electropolishing.

Initial experiments with a levelling type of bright-nickel solution indicated that the thickness of nickel required would be such as to render the process commercially uneconomic for finishing zinc-base die castings. Attention was therefore directed to obtaining the required levelling action by means of copper or zinc undercoatings on which a normal thickness of nickel could subsequently be deposited. The procedure ultimately developed comprises deposition of 0·001-0·002 in. (0·025-0·5 mm.) of zinc, from a levelling solution (see composition, p. 10), followed by 0·0001-0·0003 in. (0·0025-0·0075 mm.) of copper and 0·0005-0·0008 in. (0·0125-0·02 mm.) of bright nickel. The degree of levelling obtained by the use of the zinc solution is illustrated by photomicrographs of typical surfaces.

Copper is deposited from a cyanide solution, and the nickel-plating solution is of the sulphate/chloride type, containing wetting agents, and brighteners of unspecified composition.

Zinc-Plating Solution

	g./L.
$ZnSO_4.7H_2O$	450
NH_4Cl	15
H_3BO_3	15
Sodium citrate	25
Citric acid	25
Na_2SO_4	15
Glycerin	7
Saccharin	18
p-Toluene sulphonamide ..	2·7
Tergitol, by vol.	0·5%
pH .. 2·4±0·05	

Operating Conditions

Periodic Reverse cycle

Direct	15 secs.
Reverse ..	9 secs.

(The current density during the reverse part of the cycle is about 5 per cent. higher than that used during the direct stage.)

Agitation: Air+work bar
Temperature: 115°-118°F. (46°-48°C.)
Anodes: Special high-grade zinc slabs bagged in nylon.

A further section of the investigation on plated zinc-base die castings comprised study of the mechanism of blistering and corrosion and the development of improved plating techniques for prevention of such defects. A significant observation in this work was the finding of large numbers of surface pits which penetrated only part of the thickness of the nickel coating. The evidence found confirmed the concept that corrosion usually begins in the nickel layer, under pits or cracks in the outer coating of chromium. The nickel is more active galvanically than the chromium, and pitting continues until the nickel is completely penetrated. (The accelerating effect of faulty chromium plating on pitting in nickel has previously been reported; *ibid.*, 1958, vol. 45, pp. 144-50: *Nickel Bulletin*, 1958, vol. 31, No. 5, p. 130.) The observations made during this later research, combined with the earlier data, indicate that the use of improved chromium coatings, free from pits and cracks, would be the most promising means of securing better corrosion-resisting qualities in plated die castings. Other possibilities were, however, also explored, and the programme included combinations of plating which would demonstrate the effects of (1) increasing the thickness of the copper and the nickel layers, (2) interposing levelling zinc plate between the zinc basis metal and the copper coating, and (3) replacing conventional chromium with thicker chromium deposited in crack-free form. The panels so treated were exposed to normal atmospheric corrosion tests and to evaluation by two accelerated tests: (1) the acetic-acid/salt-spray test, and (2) the 'Corrodkote' test.

Under all the test conditions the performance of the bright crack-free chromium, in improving corrosion-resistance, was the most outstanding feature. Increasing the thickness of copper and of nickel was less effective than increasing the thickness of chromium. The performance of panels plated from the levelling zinc solution is reported as 'encouraging', and it is considered that complex-shaped castings such as are used in the automobile industry would probably benefit even more than the flat test panels from such treatment.

In view of the success achieved with crack-free chromium coatings, further work is being done on the influence of thickness of chromium, and, among other variables, the behaviour of duplex nickel undercoatings is being evaluated.

'Composite Electrochemical Materials': Electrodeposition of Carbide/Metal Composites

'Carbide Co-Deposition: A New Hard-Plating Technique.'

Metalworking Production, 1958, vol. 102, Aug. 29, p. 1514.

The note refers to a new electrodeposition process, developed by Platecraft, Inc., Buffalo and designated 'Composite Electrochemical Materials', which produces thin composite coatings of finely-divided particles of hard carbides or oxides in a metallic matrix. The technique, which is claimed to be no more complex than methods used in conventional plating, provides an economical method of obtaining coatings combining the strength and ductility of the metallic constituent with the abrasion-resistance conferred by the carbide or oxide.

Any metal that can be electrodeposited is suitable for use with the process: the carbides of silicon, tungsten, titanium and chromium, the oxides of aluminium or zirconium, and the metals molybdenum or tungsten can, for example, be co-formed with such basis metals as nickel, cadmium, silver and copper. A coating produced by co-deposition of silicon carbide and nickel to a thickness of only 0·001 in. (0·025 mm.) is claimed to double the life of sawblades. Applied to tools and gauges, the coatings have given five times the life of hard-chromium deposits, and the service life of dies used in forging alloy steels has also been improved. In the manufacture of bearings a composite of silicon carbide and silver is said to exhibit eight times the wear-resistance of pure silver, while coatings with nickel as the metallic constituent have the additional advantage of increased oxidation- and corrosion-resistance.

In the atomic energy field the technique enables uranium and thorium oxides or carbides to be dispersed in nickel, iron or cadmium, and may provide a means of preparing mixtures of rare-earth oxides and cadmium or iron with greater control over particle distribution. 'Composite Electrochemical Materials' are expected to find application in aircraft and missile manufacture (e.g., turbine blades, skin surfaces) and car production (engine parts, clutch discs, etc.), but their main potential is in the manufacture of such household articles as knives, scissors, and garden and home-workshop tools.

Electrodeposition of Molybdenum and Molybdenum Alloys

D. W. ERNST and M. L. HOLT: 'Cathode Potentials during the Electrodeposition of Molybdenum Alloys from Aqueous Solutions.'

Jnl. Electrochemical Soc., 1958, vol. 105, Nov., pp. 686–92.

Although molybdenum readily co-deposits with iron, cobalt and nickel from aqueous solutions, it has not yet been found possible to deposit appreciable amounts of pure molybdenum in such conditions. The work reported here was carried out in an attempt to explain this cathodic behaviour of molybdenum.

In view of the failure of other methods of approach, cathode-potential measurements were used as the mode of investigation. Potentials were measured by the direct method, during the electrolysis of aqueous ammoniacal citrate solutions containing sodium molybdate and the sulphate of a co-depositing metal (iron, nickel or cobalt). The results indicate that the reduction of molybdate ion in this type of bath is probably not accomplished in one step with six electrons, but with one, two, or three electrons, depending on the metal being co-deposited and its state of oxidation in the solution. It is also indicated that hydrogen is involved in the molybdate-reduction process. The authors propose a two-step mechanism for the reduction of molybdate ion in the presence of a co-depositing metal, and suggest an explanation of why this metal must be iron, cobalt or nickel rather than such metals as chromium, manganese, copper or zinc.

NON-FERROUS ALLOYS

Revised A.S.T.M. Specifications for Nickel Alloys

See abstract on p. 3.

Interdiffusion of Aluminium and Nickel

See abstract on p. 4.

Physical Properties of 'Nichrome' Evaporated Films

D. W. MOORE: 'Study of Physical Characteristics of Thin-Film Resistance Elements.'

Wright Air Development Center, Tech. Report 57-371, 1957; 41 pp. *P.B.* 131703.

The application of metal-evaporation techniques to production of elements for electrical components is assuming increasing importance in the electronics industry. A major disadvantage of the technique, however, is the large and inexplicable variation which is occasionally encountered in the properties of films deposited under similar conditions of evaporation. In the work described in this report the properties of such films were studied, in an attempt to throw light on the phenomenon by determination of the relationship between their structure and electrical characteristics.

Three materials were investigated: the 80–20 nickel-chromium alloy 'Nichrome', a nickel-titanium alloy, and carbon. Practically all the work was carried out on the 'Nichrome' alloy, though some data on nickel-titanium and carbon films are reported.

The stability of the films was studied by two methods:

(1) Metallographic examination, by means of X-ray diffraction and electron microscopy, of resistance films at various stages in their manufacture.

(2) Determination and interpretation of resistance and temperature coefficient of resistance. Measurements were made on resistors which had been withdrawn during vacuum annealing, and investigation of the rôle of the substrate was made on specimens which had been evaporated on 'Vycor' instead of on ceramic. Other specimens studied included: a series of finished products subjected to the same processing treatments; specimens having about one hundredth of the resistance per square of the regular samples, and others having the same low resistance but deposited on smooth rather than rough ceramic. Samples from these groups were subjected to various treatments (e.g., oxidation, reduction in hydrogen, treatment with lithium) in an attempt to learn something of the structure of the films by studying their effects on resistance and temperature coefficients. Information on the influence of strain on resistance changes was obtained by measuring resistances of specimens under pressure.

The results obtained indicate that 'Nichrome' films are composed of metallic regions bounded by areas of oxide. The films are in a condition of some strain, which causes periodic and discontinuous increases in resistance: the more gradual increase observed in resistance is due to slow oxidation. The ageing curves, temperature coefficients of resistance, effects of humidity, reduction, oxidation and introduction of lithium, and measurements made at high current densities or under pressure can all be interpreted in terms of these factors. The condition of strain is attributed to the considerable difference between the coefficients of linear expansion of 'Nichrome' and of the ceramic substrate (the alloy has a very high coefficient which cannot be matched by that of any available ceramic or glass). Recommendations are made with respect to methods of minimizing such differences in coefficient of expansion and of combatting the problem of oxidation.

Magnetic Properties and Structure of Nickel-Manganese Alloys

R. HAHN and E. KNELLER: 'Magnetic Properties and Ordered Structure of Nickel-Manganese Alloys. II. Change in Properties of Single Crystals resulting from Plastic Deformation.'

Zeitsch. f. Metallkunde, 1958, vol. 49, Sept., pp. 480–8.

Report of study of the plastic behaviour of nickel-manganese single-crystal alloys, and of the influence of deformation and subsequent recovery, on the

magnetic and electrical properties of the alloys. The materials studied contained 22·7, 23·7, 24·6 and 32·9 at. per cent. of manganese.

The results indicate that the plastic behaviour of the alloys, in both ordered and disordered conditions, shows a general resemblance to that observed in other face-centred-cubic alloys in which an ordering phase of the $AuCu_3$ type occurs. *Inter alia*, the following observations are made:

Critical shear stress is considerably higher in an ordered than in a disordered crystal. Coercive force increases with plastic deformation: in crystals containing less than 25 at. per cent. manganese the increase is small, whereas in alloys containing higher percentages of manganese the increase produced by deformation is considerable. The reasons for these effects and for the influence of thermal recovery on magnetic and electrical properties are discussed.

Spontaneous Magnetization in Nickel-Copper Alloys

S. A. AHERN, M. J. C. MARTIN and W. SUCKSMITH: 'The Spontaneous Magnetization of Nickel+Copper Alloys.'

Proc. Royal Soc., Ser. A, 1958, vol. 248, Nov. 11, pp. 145–52.

The paper describes measurements of the spontaneous magnetization of some nickel-copper alloys of high purity, containing up to 54·11 at. per cent. of copper. In all cases measurements were made over the temperature range Curie point down to 80°K. (−193°C.) and for alloys containing more than 30 at. per cent. of copper the determinations were extended to 23°K. (−250°C.).

Elastic Modulus of Nickel-Copper Alloys

See abstract on p. 4.

Grain Size in Cast Alloys: Delineation, Estimation and Significance

See abstract on p. 1.

Intergranular Cavitation in Stressed Copper-Nickel Alloys

B. J. REID and J. N. GREENWOOD: 'Intergranular Cavitation in Stressed Copper-Nickel Alloys.'

Trans. Metallurgical Soc., Amer. Inst. Mining and Metallurgical Engineers, 1958, vol. 212, Aug., pp. 503–7.

Previous investigations by GREENWOOD, MILLER and SUITER had shown that intergranular cavitation occurs in copper and 70-30 brass as a result of tensile stressing at elevated temperatures (see *Acta Metallurgica*, 1954, vol. 2, Mar., pp. 250–8). It was found that, for a given rate of stress, cavities formed in the brass at a lower temperature than in copper, and were, moreover, larger, and it was thought that this variation might be attributable to the distortion produced in the metal lattice by the alloying element. If the cavitation is caused by the vacancy-migration mechanism proposed by Greenwood, the distortion of the lattice would reduce the energy required for formation of vacancies, and thus facilitate onset of cavitation. It has also been suggested that cavities may be nucleated by migration of hydrogen atoms to the less regular atomic spacing at the grain boundaries: there they combine to form molecules which, being too large to diffuse back into the lattice, form stable cavities into which vacancies could condense. The work reported was carried out to determine the effect of the presence of an alloying element on the occurence of intergranular corrosion in copper, i.e., to determine, in tests on a series of copper-nickel alloys, whether increase in the nickel content would result in a corresponding increase in, or modification of, the cavities formed. Nickel was selected as the alloying element for two reasons: (1) it forms solid solutions with copper throughout the whole range of alloys; (2) its vapour pressure is low and enables the hydrogen content of the alloys to be minimized by vacuum melting.

Tests were made on high-purity copper and copper-nickel alloys containing, after melting, 5·7, 10·8, 21·7, 33·7 and 43·7 per cent. of nickel, respectively. The specimens were cold-rolled to strip 0·03 in. (0·75 mm.) thick, and homogenized *in vacuo* for one hour at 900°C. after each 30 per cent. reduction. At a thickness of 0·05 in. (1·25 mm.) the strip was held for 24 hours at 450°C. in an attempt to reduce the hydrogen content still further. Prior to testing, specimens were annealed for two hours at 800°C.

Tensile tests were carried out *in vacuo*, at temperatures ranging from 20° to 650°C. Ductility curves so obtained are presented. A detailed examination was made of the progress of surface deformation in the 10 per cent. nickel alloy, and the degree of deformation required to initiate visible cavitation was determined on the 20 per cent. nickel alloy.

The main results are summarized as follows:

'(1) At some temperature during deformation under a constant strain rate of 1·7 per cent. per hour, all the alloys of copper containing up to 44 at. per cent. of nickel exhibited the phenomenon of intergranular cavitation. The cavities appeared preferentially in the boundaries normal to the tensile stress.

'(2) No obvious difference was determined between the degree of cavitation exhibited by copper in the as-received or vacuum-melted condition.

'(3) In general, the number and size of the cavities increased with increase in nickel content up to 20 per cent. and then markedly decreased.

'(4) The higher the nickel content, the higher the temperature at which cavitation became visible. For pure copper, cavities were visible (× 500) in the grain boundaries after straining at 200°C.: for alloys containing 20 per cent. nickel, at 250°–300°C. : and for alloys containing 30–40 per cent. nickel, at 500°C.

'(5) The type of fracture changed from trans- to inter-crystalline as the temperature of straining was raised. With a nickel content

of 10 per cent. or more, however, and under conditions when grain-boundary migration occurs, fracture was found to be a mixture of the two types.

'(6) For alloys containing 10 per cent. and more nickel, fall in ductility with rise in temperature was arrested at 400°–500°C. With a content of 10 and 20 per cent. nickel there was a marked recovery, followed by a further rapid decrease above 600°C. This latter effect was most pronounced with the 10 per cent. alloy and scarcely evident with the 30 and 40 per cent. alloys.

'(7) Recovery of ductility was associated with grain-boundary migration on a micro scale and with recrystallization. It appears that formation of intercrystalline cracks from strings of cavities is arrested by the migration. When the temperature was sufficiently high (600°C.), the grain boundaries remained smooth and strings of cavities again united to form cracks.

'(8) The higher the nickel content, the higher the temperature at which grain-boundary migration appeared.

'(9) The higher the temperature, the less was the deformation needed to induce cavitation in any given alloy.

'(10) In the case of the 20 per cent. alloy, cavitation began (at the rate of stress used in the tests) as the mode of deformation changed from slip to grain translation, i.e., in the temperature range 250°–400°C. The effect was then complicated by grain-boundary migration as the temperature was further raised.'

Although cavitation was not distinguishable in the alloys containing 30 and 40 per cent. nickel at temperatures below 500°C., it is considered that the loss in ductility is an adequate indication of the presence, at the grain boundaries, of cavities too small to be visible. It would then appear that a nickel content higher than 20 per cent. renders the formation of cavities difficult.

Erosion and Corrosion of Nickel-containing Alloys in Wet Oxygenated Steam

See abstract on p. 21.

Compatibility of Nickel and Nickel-containing Alloys with Graphite at Elevated Temperatures

See abstract on p. 20.

Composition Range of Ti_2Ni

G. A. YUKO, J. W. BARTON and J. GORDON PARR : 'The Composition Range of Ti_2Ni.'

Trans. Metallurgical Soc., Amer. Inst. Mining and Metallurgical Engineers, 1958, vol. 212, Oct., pp. 698–700.

Although previous work has shown the composition range of the complex face-centred-cubic phase Ti_2Ni to be narrow, no precise limits had previously been established. The work reported in this paper consisted in preparation of homogeneous alloys on either side of the stoichiometric composition (33·33 at. per cent.) and determination, by optical and X-ray-diffraction metallographic techniques, of the composition limits of the phase. A series of alloys containing 20–45 per cent. nickel, in 5 per cent. increments, was prepared, by levitation melting from iodide titanium and spectrographic-standard nickel. Methods used for preparation and examination of the specimens are described.

Measurements on specimens of the respective alloys lead to the following conclusions: 'Within the limits of accuracy of the X-ray-diffraction technique used, the lattice parameter of Ti_2Ni is the same on both sides of the stoichiometric composition. Therefore, if Vegard's Law is assumed, a range of composition of not more than 2 per cent. on either side of the stoichiometric (at and below 700°C.) is indicated.

'Microscopical metallography shows that single-phase structures are produced by quenching from 700°C. alloys containing ½ per cent. nickel more than, or less than, stoichiometric composition. Alloys containing 1 per cent. more than, or less than, stoichiometric composition show a two-phase structure. It is therefore concluded that at 700°C. the composition limits for Ti_2Ni lie between ½ and 1 per cent. nickel on either side of the stoichiometric composition.

'The fact that annealed alloys containing ½ per cent. nickel more than, or less than, stoichiometric composition show two-phase structures indicates that the composition range of Ti_2Ni narrows with decreasing temperature.'

Methods for Measurement of Thermal Conductivity of Solids

See abstract on p. 2.

Gravimetric Separation of Copper, Nickel and Zinc

See abstract on p. 6.

NICKEL-IRON ALLOYS

Methods for Measurement of Thermal Conductivity of Solids

See abstract on p. 2.

CAST IRON

Grain Size in Cast Iron: Delineation, Estimation and Significance

See abstract on p. 1.

Erosion and Corrosion of Nickel-containing Cast Iron in Wet Oxygenated Steam

See abstract on p. 21.

CONSTRUCTIONAL STEELS

Effect of Cadmium Plating on the Tensile Properties of Nickel-Chromium-Molybdenum High-Strength Steel Stressed at High Temperatures

E. M. KENNEDY: 'The Effect of Cadmium Plating on S.A.E. 4340 Steel in the Presence of Stress Concentrations at Elevated Temperatures.'

Wright Air Development Center, Tech. Report 58-108, *Part I*, Mar. 1958; 34 pp. *P.B.* 131814.

Previous investigations in connexion with the use of high-strength steels for aeronautical applications have shown that such materials are subject to design criteria which vary in many respects from those obtaining at lower strengths (see, for example, abstracts in *Nickel Bulletin*, 1958, vol. 31, No. 8, pp. 225-6, which include reference to the effect of cadmium plating on susceptibility to hydrogen embrittlement). Aircraft components fabricated from high-strength steels are frequently protected from corrosion by electrodeposition of cadmium. Experience has shown, however, that the tensile strength of the coated steels decreases sharply when the components are exposed under stress at elevated temperatures: failures by brittle fracture have occurred in service at loads corresponding to stresses well below the tensile strength of the steel, and deposits of cadmium have been found on the fractured surface.

The work now described was carried out to obtain information on the effect of cadmium plating on the high-temperature tensile properties of a low-alloy steel heat-treated to various levels of strength.

The steel was of the following composition: carbon 0·40, silicon 0·22, manganese 0·82, sulphur 0·015, phosphorus 0·012, nickel 1·60, chromium 0·85, molybdenum 0·27, per cent. Stress-rupture properties were determined at 500°, 600° and 611°F. (260°, 315° and 321°C.), on specimens in the annealed condition and on others which had been heat-treated to the intermediate tensile-strength range or to achieve tensile strengths of 240,000-260,000 p.s.i. (106-116 t.s.i.; 169-183 kg./mm.^{2}). The tests were made on notched and un-notched specimens, plated and unplated.

The results show that the tensile properties of the steel in the annealed condition were unaffected at 600° or 611°F. by cadmium plating. Plating the steel in the heat-treated condition (i.e., showing tensile strengths in the 240,000-260,000 p.s.i. range) resulted, however, in marked decrease in rupture strength at temperatures at or near the melting point of cadmium (609·6°F.; 321°C.). Exposure of unstressed cadmium-plated specimens to these temperatures caused no decrease in the room-temperature tensile strength. Further work is due to be carried out on the fatigue and stress-rupture properties of cadmium-plated components at high temperatures.

See also following abstract

Embrittlement of High-Strength Steels Resulting from Attack by Molten Cadmium

B. COHEN: 'Cadmium Embrittlement of High-Strength Steels at Elevated Temperatures.'

Plating, 1958, vol. 45, Nov., pp. 1127-32.

A considerable literature has already accumulated on the embrittlement of high-strength steels caused by plating with cadmium (see, for example, papers abstracted in *Nickel Bulletin*, 1958, vol. 31, Nos. 1 and 2, pp. 12 and 42). The problem calls for investigation from two aspects: (1) hydrogen embrittlement arising from reactions during electroplating, and (2) the embrittling effect of molten cadmium which becomes operative when the coated steel parts are heated at temperatures above the melting point of cadmium (609·6°F.; 321°C.).

This paper reports normal- and elevated-temperature tests on smooth and notched specimens of two types of high-tensile steel, of which typical compositions are given in the table below.

Specimens of both steels, heat-treated to a high-tensile level, were cadmium-plated from a fluoborate solution and were baked at 375°F. (190°C.) for 23 hours after plating. Room-temperature tests, on smooth and notched specimens, established that no detrimental residual hydrogen embrittlement remained at the test-stress level.

The results of the elevated-temperature tests show that, at temperatures above that of the melting point of cadmium, cadmium coatings drastically lower the tensile strength of high-strength steels. The embrittling effect of the molten cadmium is instantaneous, resulting in an extremely brittle type of failure. (Typical fractures are shown, and values are given for the strength, at 620°F. (326°C.), of the bare steels and of the same materials coated with cadmium, which, at that temperature, had become molten.) The embrittling effect of the cadmium is particularly severe at points of stress concentration, and it is obvious that cadmium-plated parts should therefore not be specified for use at temperatures approaching the melting point of cadmium. An amendment has been inserted in *U.S. Federal Specification QQ-P-416a*, 'Plating, Cadmium, Electrodeposited', to read:

'Cadmium coatings shall not be used on parts

Steel	C %	Si %	Mn %	S %	P %	Ni %	Cr %	Mo %	V %
S.A.E. 4340	0·39	0·25	0·73	0·010	0·012	1·74	0·81	0·24	—
S.A.E. H-13 ('Thermold J')	0·52	1·09	0·35	0·007	0·027	—	5·08	1·40	0·99

which in service reach a temperature of 500°F. (260°C.) or higher, or come into contact with other parts which reach those temperatures.' The same restriction has been embodied in *U.S. Specification MIL-C-8837 (ASG)*, 'Coating, Cadmium (Vacuum Deposited).'

The mechanism of embrittlement by molten cadmium is not yet understood. The coating metal actually penetrates the stressed steel, as is illustrated in this report, but since iron is not soluble in molten cadmium the resulting embrittlement cannot logically be attributed to an alloying effect. The author proposes the following explanation:

(1) Stressed high-strength steel deforms at grain boundaries, leading to a higher concentration of dislocations, whilst in annealed steels the stresses are absorbed within the grains. The effect would be most pronounced at areas of stress concentration or at notches.

(2) Molten cadmium, being in a state of high activity, very rapidly diffuses into the dislocated grain boundaries.

(3) The molten cadmium in the grain boundaries interrupts the continuity of the structure and produces a condition known as 'hot shortness'. This results in intergranular cracking of the steel.

Erosion and Corrosion of Nickel-containing Steels in Wet Oxygenated Steam

See abstract on p. 21.

Influence of Residual and Minor Elements on Crack-Resistance of Welds in High-Tensile Steels

R. P. SOPHER: 'Effects of Chemical Composition on Cracking Resistance of High-Strength-Steel Weld Metals.'

Welding Jnl., 1958, vol. 37, Nov., pp. 481s-92s.

Report of research sponsored by Wright Air Development Center, in connexion with requirements of the aircraft and missile industries for welding steels which, after welding, can be heat-treated to tensile levels of the order of 250,000 p.s.i. (111·5 t.s.i.; 176 kg./mm.²).

Survey of the literature on hot cracking of welds demonstrated that weld metals which have failed from that cause provide evidence that very little, if any, straining occurs before failure. It was therefore indicated that a test designed to determine the properties of weld metals at elevated temperatures should be of value in ascertaining the temperature at which cracking occurs. The test conditions built up on this concept were accordingly planned to measure the strength and ductility of the relevant materials at various temperatures during cooling from the liquid state. This freezing-cycle, hot-tension test provides for a very high rate of cooling, simulating conditions present during the welding cycle. Full details of test conditions are given, with illustration of testing machine, specimen, etc.

The parent material used throughout was aircraft-quality steel to S.A.E. 4340 specification (carbon 0·38–0·43, silicon 0·2–0·35, manganese 0·6–0·8, sulphur 0·040 max., phosphorus 0·040 max., nickel 1·65–2·0, chromium 0·7–0·9, molybdenum 0·2–0·3, per cent.). A study was made of the influence of the following elements: carbon (0·30–0·59 per cent.), sulphur, phosphorus. In view of the known beneficial effects of rare-earth metals, on workability, the possibility of securing reduction in weld-metal cracking by such additions was also investigated.

The results confirm the observations of other researches that hot cracking of a weld deposit is related to the ductility and strength properties just below the nominal solidus temperature. If the weld deposit is not capable of deforming, and thus relieving the stresses that are present from contraction during cooling, failure will occur. This failure is intergranular; consequently, it can usually be traced to a eutectic mixture which is the last to freeze.

By controlling the composition of the low-alloy high-tensile type of steel studied, it is possible to deposit sound, crack-free welds under restrained conditions, thus permitting fabrication of welded assemblies which may be heat-treated to ultra-high-strength levels of 280,000 p.s.i. (125 t.s.i.; 197 kg./mm.²).

The test method described is capable of detecting differences in steels having minor differences in composition. The results obtained by that test give good agreement with those of the restrained-weld test which has already been used for evaluation of crack susceptibility in high-tensile steels.

With regard to composition, the results lead to the following conclusions:

Increase in carbon from 0·20 to 0·59 per cent. caused only a small deterioration in resistance to weld cracking, provided that the sulphur and phosphorus were maintained at low levels. Increase in sulphur from 0·004 to 0·030 per cent. in these weld metals resulted in loss of strength and ductility at sub-solidus temperatures, and these impaired properties were reflected in decrease in resistance to cracking as the sulphur was raised. Increase in phosphorus from 0·004 to 0·017 per cent. had little effect on the high-temperature properties of the weld metal, but further increase, to 0·039 per cent., resulted in pronounced decrease in strength and ductility at temperatures near the solidus and in poor resistance to cracking. This content of phosphorus is within normal specification limits for aircraft steels, but should be reduced for steels which are to be welded.

Addition of 3 lb./ton of 'Mischmetall' to a heat of normal sulphur content (0·03 per cent.) improved ductility and strength at temperatures near the solidus: such an addition reduced the sulphur content to 0·025 per cent. Doubling the addition of 'Mischmetall' (i.e., 6 lb./ton) resulted in further improvement in properties, by lowering the sulphur to 0·014 per cent., and by producing a more favourable type of inclusion in the steel.

HEAT- AND CORROSION-RESISTING MATERIALS

Effect of High-Temperature Ageing on Development of Minor Phases in 'Inconel X'

W. C. BIGELOW, J. A. AMY and L. O. BROCKWAY: 'The Effect of High-Temperature Ageing on the Development of Minor Phases in an Age-Hardening Nickel-base Alloy.'

Trans. Metallurgical Soc., Amer. Inst. Mining and Metallurgical Engineers, 1958, vol. 212, Aug., pp. 543-9.

Studies on relatively simple nickel-chromium alloys rendered age-hardenable with aluminium and titanium have shown that their favourable high-temperature properties are related to the precipitation of the intermetallic γ' phase having a structure based on that of Ni_3Al phase of the nickel-aluminium system. The investigation reported in this paper was initiated to identify the minor phases and to study the microstructural changes occurring in one of the more complex commercial alloys as a result of ageing in the temperature range to which it is likely to be subjected in service. Particular emphasis was placed on investigation of the rôle of the γ' phase. (In the same field, the results of an investigation of the effects of heat-treatment on microstructure and high-temperature properties of a similar alloy have been published by DECKER, ROWE, BIGELOW and FREEMAN as *National Advisory Committee for Aeronautics, Tech. Note* 4329, July 1958: see abstract in *Nickel Bulletin*, 1958, vol. 31, No. 12, p. 334.)

'Inconel X' was selected for study, since extensive data were already available on the influence of various ageing treatments on the mechanical properties of the alloy (see FREY, FREEMAN and WHITE, *National Advisory Committee for Aeronautics, Tech. Note* 2385, June 1951; *Nickel Bulletin*, 1952, vol. 25, No. 1, pp. 17-18). The present investigation thus also offered the opportunity of correlating the properties achieved with the microstructures resulting from ageing at similar temperatures.

The specimens, which had been solution-treated at 2050°F. (1120°C.) and water-quenched, were aged for 1, 10, 100 and 1000 hours at 1200°, 1400° and 1600°F. (650°, 760° and 870°C.). Microstructural changes were studied by electron microscopy, and the minor phases were identified by X-ray and electron diffraction.

Four minor phases were identified: titanium nitride, niobium nitride, a complex carbide $M_{23}C_6$, and γ'. The two nitrides were found (as large blocky particles) in all the aged specimens, as well as in the alloy in the solution-treated condition. The γ' phase formed within the matrix grains at all three ageing temperatures. The $M_{23}C_6$ phase, which precipitated within the grain boundaries, occurred only after ageing at 1200° and 1400°F. (650° and 760°C.).

FREY *et al.* found that hardness, creep strength at high stresses, rupture strength at low stresses, yield strength and internal strain were all governed by precipitation of minor phases within the matrix. Comparison of the electron micrographs obtained by the present authors with the data obtained by FREY showed that the ageing times required to achieve an improvement in these properties are approximately the same at all temperatures as those required to produce detectable γ' particles in the alloy, and that the properties continue to improve during the early stages of particle growth. Optimum properties during ageing at 1400° and 1600°F. (760° and 870°C.) occurred as the γ' particles began to coalesce and to depart from the condition of critical dispersion. Continued ageing results in further growth of the particles and a deterioration in properties. Correlation of the size and distribution of the γ' particles also confirmed the importance of these factors in relation to mechanical properties.

The finding that optimum properties are obtained when the γ' phase is sufficiently developed to be distinguishable by electron microscopy is considered of significance, since it suggests that precipitation phenomena in such alloys can be satisfactorily investigated by metallographic techniques.

Boron and Zirconium in High-Temperature Alloys

R. F. DECKER and J. W. FREEMAN: 'Mechanism of Beneficial Effects of Boron and Zirconium on Creep-Rupture Properties of a Complex Heat-Resistant Alloy.'

Nat. Advisory Committee for Aeronautics, Tech. Note 4286, Aug. 1958; 54 pp.

Recent investigations have demonstrated the value of trace amounts of boron and zirconium as affecting the creep-rupture properties of nickel-base high-temperature alloys: see, for example, papers abstracted in *Nickel Bulletin*, 1958, vol. 31, Nos. 5 and 6, pp. 137 and 170. One of the earlier investigations indicated that even traces of these elements introduced inadvertently from contamination with crucible materials were of significance. In the interim the usefulness of the addition elements has been verified on a number of nickel-base alloys and their effects on alloys of other systems have also been considered.

Since the beneficial influence of zirconium and boron thus appears to be operative in a range of alloys, it was considered advisable to study the mechanism of the changes effected by such additions. The material chosen was an alloy containing nickel 55, chromium 20, cobalt 15, molybdenum 4, titanium 3, aluminium 3, per cent., selected because the improvement of properties through zirconium and boron additions had already been established on such an alloy. As an introduction to the report, a short survey is presented of current views on the rôle of the γ' and carbide phases in nickel-base alloys hardened with titanium+aluminium, into which group this alloy falls.

The general programme of investigation comprised observations of microstructural changes produced by varying amounts of zirconium and boron, when

the alloy was subjected to thermal treatments involving elevated temperatures and stress. The amounts present, used singly or in conjunction with one another, were boron 0·0002–0·0090, zirconium <0·001–0·19, per cent.

The observations made, which are recorded in detail, confirm earlier findings with regard to the improvement in creep-rupture properties associated with additions of boron and zirconium. The authors conclude that it results from the stabilizing influence which these elements exercise on the grain boundaries of the material. The alloy of very low boron and zirconium content was subject to rapid agglomeration of $M_{23}C_6$ and γ' in the grain boundaries, followed by depletion of γ' and intergranular micro-cracking at the grain boundaries transverse to the applied stress. Brittle fracture then ensued by joining up of micro-cracks.

Zirconium, boron, and boron+zirconium retarded such changes. The relative effectiveness of the three types of addition increased in the order listed, thus allowing longer life to higher deformations before fracture occurred. In the absence of boron and zirconium extensive micro-cracking was observed early in the second-stage creep period, at relatively short times, and fracture occurred prematurely, with very little deformation. Suitable amounts of boron+zirconium delayed occurrence of micro-cracking until after third-stage creep had started; thus creep-rupture life was prolonged and ductility-to-fracture was markedly increased.

There was no indication that the two addition elements influenced the size, amount and distribution of the intragranular γ' phase, and it is concluded that the beneficial effects on properties did not result from any change in the intragranular-γ' reaction.

Discussion of the mechanism of the stabilizing influence of boron and zirconium, on the grain boundaries, leads to the conclusion that the explanation which best accords with the observed effects is that of retardation of agglomeration by preferential segregation of boron and zirconium to grain boundaries.

The possible theories relating to the mechanism of the action of the trace elements (and other aspects of the subject) are discussed in relation to published information. The report includes a bibliographical survey of relevant data from 44 *items of literature*.

High-Temperature Properties of Nickel- and Cobalt-base Alloys Melted and Cast in Air and *in Vacuo*

M. J. STUTZMAN: 'Studies and Comparison of the Properties of High-Temperature Alloys Melted and Precision Cast both in Air and in Vacuum.'

Wright Air Development Center, Tech. Report 57-678, Mar. 1958; 103 pp. *P.B.* 131807.

Recent developments in the technology of vacuum melting have focused attention on its potential as a means of producing high-temperature alloys with improved properties. Investigations in this connexion have, however, produced conflicting results, which in many cases have been confused by the other variables involved. The test programme described in this report was initiated in an attempt to determine the influence of melting atmosphere (air or vacuum) on the stress-rupture and tensile properties and oxidation behaviour of four cast commercial heat-resisting alloys.

Two nickel-base and two cobalt-base alloys were selected for study. Their compositions are given in the table below.

The melting and casting methods by which the alloys were finally produced may be summarized as follows:

'Stellite 31' and 'He 1049': (1) melted and investment-cast in air; (2) melted *in vacuo* and investment-cast in an argon atmosphere.

'Utimet 500': melted and cast *in vacuo*.

'Guy's Alloy': prepared either *in vacuo* or in an argon atmosphere.

Initial tests on the two nickel-base alloys, in the air-melted condition, showed them to have consistently inferior properties. The programme was therefore revised, with respect to these alloys, (1) to study the influence of boron and zirconium additions on the properties of vacuum-melted specimens of 'Utimet 500' containing from 0·11 to 0·15 per cent. of carbon, and (2) to compare the properties of vacuum- and argon-melted specimens of 'Guy's Alloy'.

The specimens were evaluated by: (1) Stress-rupture tests, at 1350°, 1500°, 1600°, 1700° and 1800°F. (730°, 815°, 870°, 925° and 980°C.), with the aim of obtaining data for 100 and 250 hours; (2) tensile tests at room temperature, 1200° (650°C.) and 1500°F. (815°C.); (3) oxidation tests on specimens exposed for

Alloy	Ni %	Co %	Cr %	Fe %	W %	Mo %	Mn %	Si %	C %	B %	Al %	Ti %	Nb %
'Stellite 31'	10·0	bal.	25·5	2 max.	7·5	—	0·8	0·8	0·5	—	—	—	—
'He 1049'	10·0	bal.	26·0	2 max.	15·0	—	0·8	0·8	0·5	0·4	—	—	—
'Utimet 500'	bal.	15·0	17·0	4 max.	—	4·0	0·75	0·75	0·11-0·15	—	3·0	3·0	—
'Guy's Alloy'	bal.	—	15·0	5 max.	—	5·5	0·5	0·5	0·12-0·15	0·5	6·0	—	2·0

100, 200 and 400 hours at 2000°F. (1095°C.), or subjected to thermal cycling every hour in the range room temperature to 2000°F. (1095°C.); (4) metallographic examination, gas analyses, etc. Full details are given of the melting, casting and test procedures used.

The data obtained for each alloy are presented *in extenso*. The salient findings are summarized below.

Tensile Properties

The ultimate strength of 'Stellite 31' in the air-melted condition was somewhat the higher, except at 1500°F. Vacuum-melted specimens exhibited appreciably higher elongation at all three test temperatures.

No appreciable variation was found between the properties of vacuum-melted and air-melted 'He 1049'.

At room temperature and at 1200°F. (650°C.) the ultimate tensile strength of 'Utimet 500' without boron or zirconium additions was higher than that of the modified alloy. The heat containing the addition elements showed greater elongation at 1700°F. (925°C.), though no appreciable difference in elongation was found at room temperature, 1200° and 1500°F. (650° and 815°C.).

The argon-melted specimens of 'Guy's Alloy' showed slightly higher ultimate strength than those vacuum-melted.

100-Hour Rupture Strength and Elongation

Vacuum-melted specimens of 'Stellite 31' were found to have slightly higher rupture strength and appreciably greater elongations at all temperatures except 1350°F. (730°C.), where rupture strength was somewhat higher for the air-melted test pieces.

The rupture strength of 'He 1049' proved to be essentially the same in specimens from both heats, but, except at 1350°F., where values were similar, elongation of vacuum-melted specimens was double that of the air-melted.

Addition of zirconium and boron to vacuum-melted 'Utimet 500' resulted in increased rupture strength, especially at the lower elevated temperatures; elongation was approximately doubled.

Neither rupture strength nor elongation of 'Guy's Alloy' varied greatly with variation in melting atmosphere.

Oxidation-Resistance

Gain in weight of vacuum-melted 'Stellite 31' specimens, both as a result of exposure for 100 and 200 hours at constant temperature and during thermal cycling, was greater than that of the air-melted heat.

Air-melted 'He 1049' exhibited greater weight gains than did the vacuum-melted alloy during the 400-hour exposure at constant temperature, and less during exposure for the same period to thermal cycling. Weight gains for an exposure time of 200 hours were practically the same for both heats.

The specimens of 'Utimet 500' containing zirconium and boron were found to have suffered greater gains in weight at all exposure times than those to which no additions had been made.

Vacuum-melted 'Guy's Alloy' exhibited the greater oxidation-resistance in both constant and intermittent tests: weight gain was exceptionally low for the constant-temperature test.

Gas Content

Vacuum melting appreciably decreased the oxygen and nitrogen contents of the cobalt-base alloys, but had no definite effect on the nickel-base 'Guy's Alloy'. The oxygen content of the vacuum-cast specimens of 'Utimet 500' containing zirconium and boron additions was lower, and the hydrogen and nitrogen contents were higher, than those of corresponding specimens without such additions.

It is concluded that no general relationships can be drawn from the data obtained: improvement in one property effected by production *in vacuo* was usually accompanied by a decrease in another. Each alloy should be tested under the specific conditions to which it would be subjected during service. Improvements obtained are usually relatively small, and contingent on close control of the variables involved.

Effect of Atmosphere on Creep-Rupture of a Nickel-Chromium-Aluminium Alloy

P. SHAHINIAN and M. R. ACHTER: 'The Effect of Atmosphere on Creep-Rupture Properties of a Nickel-Chromium-Aluminium Alloy.'

U.S. Naval Research Laboratory, Rept. 5133, May 6, 1958; 13 pp. *P.B.* 131735.

A summary of the authors' extensive investigations of the influence of environment on the creep properties of metals and alloys was given in *Report of U.S. Naval Research Laboratory Progress*, Apr. 1958 (the salient features of which were abstracted in *Nickel Bulletin*, 1958, vol. 31, No. 11, p. 296). The results of tests on nickel and 'Nichrome V' had been published separately. The present report relates specifically to the other material studied.

Elevated-Temperature Properties of High-Chromium Steels

AMER. SOC. TESTING MATERIALS: 'Report on Elevated-Temperature Properties of Chromium Steels (12-27 per cent.).'

A.S.T.M. Special Tech. Publn. No. 228; 113 pp. Published by the Society, 1958. Price $4.25.

The publication is one of a series of reports on the elevated-temperature properties of various types of material, prepared under the auspices of the A.S.T.M.-A.S.M.E. Joint Committee on Effect of Temperature on the Properties of Metals. (The titles of earlier reports are listed and reference is made to other projects on which information is likely to be published in the near future.)

The data presented (in tabular and graphical form) were obtained from research laboratories, commercial

publications, and the technical literature: sources are given on each data sheet. The report includes summary curves showing tensile strength; 0·2 per cent. yield strength; percentage elongation and reduction of area; stresses to produce rupture in 100, 1,000, 10,000 and 100,000 hours; and stresses for creep rates of 0·0001 and 0·00001 per cent. per hour (1 per cent. in 10,000 and 100,000 hours). Information is given on the heat-treatment to which the respective specimens have been subjected.

The chromium contents of the 23 steels covered the range 12–27 per cent. The steels are divided, according to the principal alloying elements, into the following groups:

12% Chromium
12% Chromium-2% Nickel
13% Chromium-2% Nickel-3% Tungsten
12% Chromium-3% Tungsten
12% Chromium-Niobium
12% Chromium-2·75% Molybdenum-Vanadium
12% Chromium-2·5% Tungsten-Vanadium
12% Chromium-5% Cobalt-3% Tungsten-Vanadium
13% Chromium-Tungsten-Molybdenum-Vanadium
12% Chromium-2% Tungsten-2% Molybdenum-Vanadium
12% Chromium-Molybdenum-Niobium-Vanadium
14% Chromium
16% Chromium-2% Nickel
17% Chromium
18% Chromium
27% Chromium
27% Chromium-Molybdenum

Miscellaneous Steels
Chromium-Titanium
Chromium-Aluminium
Chromium-Molybdenum-Aluminium
Chromium-Molybdenum
Chromium-Molybdenum-Nickel
Chromium-Tungsten

Rapid-Heating Tests on Aircraft Materials

J. B. PRESTON, W. P. ROE and J. R. KATTUS: 'Determination of the Mechanical Properties of Aircraft Structural Materials at Very High Temperatures after Rapid Heating.'

Wright Air Development Center, Tech. Report 57-649, Jan. 1958; 187 pp.

This very comprehensive report is a further contribution to the literature relating to structural components in high-speed aircraft and missiles, which are called upon to function for short periods at high temperatures and high stresses.

Mechanical properties (tensile, creep, fracture, compression, shear and bend) were investigated under conditions of moderate-to-rapid rates of heating and loading. The materials included in the tests were electrolytic tough-pitch copper, O.F.H.C. copper, nickel, ingot iron, molybdenum, tantalum, graphite and composite O.F.H.C. copper/austenitic stainless-steel sheet. Test temperatures ranged from room temperature to the melting points of the respective metallic materials, and to 5750°F. (3177°C.) for graphite. Total heating, holding, and loading times to failure varied from about 3 seconds to about 30 minutes. Many of the tests were carried out both in air and in inert atmospheres.

The mechanical strength of the metallic materials was progressively lowered by rising temperature: the strength of graphite increased up to 4500°F. (2482°C.) and afterwards decreased with further rise in temperature. Under constant-load and constant-temperature conditions, or with transient load or transient temperature, the inherent load-carrying capacity of all the metals increased with decreasing time under load.

Within the time limitations of the tests made, the mechanical properties of copper and of nickel were not significantly affected by exposure to air at temperatures up to their melting points. Oxidation deleteriously influenced the mechanical properties of the iron, molybdenum, tantalum and graphite.

Bonding of Stainless Steels with Heat-Resisting Ceramic Adhesives

H. G. LEFORT and D. G. BENNETT: 'High-Temperature-Resistant Ceramic Adhesives.'

Jnl. Amer. Ceramic Soc., 1958, vol. 41, Nov. 1, pp. 476–82.

Organic adhesives used to produce metal-to-metal bonds retain their strength only up to temperatures in the region of 400°F. (205°C.). The development of high-speed aircraft has, however, confronted the engineering designer with the problem of aerodynamic heating involving much higher temperatures, and interest has turned to adhesives of ceramic type as a means of obtaining heat-resisting bonds in honeycomb-sandwich and other structural components of aircraft. In addition to satisfactory high-temperature strength over long periods, suitable adhesives for aeronautical applications are required to have good resistance to thermal shock and to moisture, and a maturing temperature as low as possible. In this paper the authors give details of an investigation designed to develop ceramic adhesives of the glassy-bond type, suitable for joining components fabricated from chromium-nickel stainless steels of 18-8 (A.I.S.I. Type 302) and '17-7 P.H.' grades, and intended for service in the temperature range 500°–1000°F. (260°–540°C.).

The adhesives were evaluated by determination of the shear strengths of overlap joints produced in stainless-steel sheet 0·047–0·060-in. (1·175–1·5-mm.) thick. Tests were made over the range room temperature – 1000°F. (540°C.).

The basic ceramic used, designated 'UI 117-50', was of the following composition (parts by weight): potash feldspar 34·8, borax 9·2, soda ash 16·5, soda niter 4·2, barium carbonate 0·7, whiting 8·0, zinc oxide 9·2, sodium silicofluoride 4·2, $BaO.P_2O_5$ 6·9, 88% vanadium pentoxide 2·5, aloxite 3·8. The slip composition consisted of 100 parts of this frit to 2 parts of colloidal silica and 50 parts of water. When applied to the stainless steels, the adhesive was fired at 1750°F. (955°C.), a temperature particularly appropriate for use with the '17-7 P.H.' grade, since firing could be effected simultaneously with one stage of the heat-treatment used to produce the

age-hardened condition RH-950 in which the steel was tested.

The effects of variation in the thermal expansions of the adhesive and basis metal were investigated by use of porcelain-enamel adhesives on ingot-iron specimens. It was found that when the expansion of the adhesive approached that of the ingot iron shear strengths were high; if, however, the thermal expansions approximated too closely, the shear strength decreased, i.e., the thermal expansion of the adhesive must be such as to maintain the adhesive in an effective state of stress.

In the initial tests on the 18-8 stainless steel, joints with adhesive 'UI 117-50' were made without the use of a space-screen carrier, and the shear strengths obtained were only moderately satisfactory. Use of a 28-mesh stainless-steel space-screen carrier with 0·004-in. (0·1-mm.) diameter wire produced a pronounced increase in the shear strength of the joints. Values for 16-gauge Type 302 stainless steel were: nearly 1000 p.s.i. (0·7 kg./mm.^{2}) at room temperature; more than 1900 p.s.i. (1·3 kg./mm.^{2}) at 800°F. (425°C.); more than 1000 p.s.i. at 900°F. (480°C.); and 440 p.s.i. (0·3 kg./mm.^{2}) at 1000°F. (540°C.). Shear strengths in the case of the 18-gauge '17-7 P.H.' steel were 1620, 1750 and 980 p.s.i. (1·1, 1·2 and 0·68 kg./mm.^{2}) at 800°, 900° and 1000°F., respectively.

Other tests involved use of the 'UI 117-50' adhesive modified by 2 or 5 per cent. additions of stainless-steel metal oxides (Fe_2O_3, NiO and Cr_2O_3), individually or in combination. The general effect of such additions was to increase shear strength at 900° and 1000°F. (480° and 540°C.), and to decrease shear strength at room temperature, 600° and 800°F. (315° and 425°C.). An addition of 2 per cent. Cr_2O_3 gave the best overall values in the temperature range studied.

The effects of introducing into the slip 5 per cent. of fine-particle powders of aluminium, copper, silicon, carbonyl iron, and carbonyl nickel were found to vary. Carbonyl iron and silicon increased the shear strength of the bond at certain temperatures, and, with copper, these additions, though not greatly affecting shear strength, may be expected to reduce brittleness and improve the impact-resistance of the bond.

Finally, honeycomb-sandwich structures of '17-7 P.H.' steel were successfully bonded with 'UI 117-50' ceramic adhesive during heat-treatment to obtain the age-hardened condition RH-950. Relatively large areas of the panels were bonded during the treatment cycle, which required resistance to the temperature extremes involved in heating at 1750°F. (955°C.), cooling to −100°F. (−73°C.) and re-heating at 950°F. (510°C.).

Applications of 'Nimonic' Alloys in Industrial Gas Turbines

''Nimonic' Materials in Non-Aircraft G.T.s.'
Oil Engine and Gas Turbine, 1958, vol. 26, Mid-Nov., p. 286.

The rôle of the nickel-chromium-base 'Nimonic' alloys in the development of aeronautical gas-turbine units is well known. This article gives a brief survey of the high-temperature characteristics and applications of those alloys of the same series which have established themselves as materials of construction for components of industrial gas turbines. In this connexion, notes are given on the uses of 'Nimonic' 75, 80, 80A, 90 and 100. An interesting feature of the article is a table listing the industrial turbines for which 'Nimonic 75' has been used for the flame tubes, exhaust assemblies, turbine inlet ducts and nozzle blades, 'Nimonic 80A' for stator and rotor blades and turbine discs, and 'Nimonic 90' for turbine discs and turbine rotor blades.

Methods for Measurement of Thermal Conductivity of Solids

See abstract on p. 2.

Compatibility of Nickel and Nickel-containing Materials with Graphite at Elevated Temperatures

A. F. GERDS and M. W. MALLETT: 'The Compatibility of a Number of Metals and Alloys with Graphite.'
Battelle Memorial Inst., Report BMI-1261, Apr. 14, 1958; 27 pp.

The operating temperature of an atomic reactor in which use is made of graphite in contact with a metallic material is limited by the degree of compatibility between the two materials, i.e., the temperature must be maintained below that at which diffusion of carbon will result in deterioration of the desired properties in the metallic part of the couple. Selection of materials which will resist carburization at high temperatures is therefore of importance. This report gives details of investigations carried out to evaluate materials which seemed likely to combine such properties with the other characteristics demanded by the specific application.

The study comprised two phases: (1) screening tests in which the materials were subjected, under pressure and in intimate contact with graphite, to a temperature of 1850°F. (1008°C.) for 250 hours; (2) tests in which the materials found most compatible were exposed for 1000 hours at 1650°F. (900°C.), and at 1850°F. (1008°C.). Compatibility was assessed on the basis of metallographic examination and hardness tests on longitudinal sections through the reaction zones. Consideration was given to the possibility of interposing some kind of diffusion barrier between the graphite and the metallic element, either in the form of a carbide layer produced on the metal surface by reaction with the graphite, or as a third metal with a low diffusion coefficient and/or a low carbon solubility.

Screening tests were made on molybdenum; copper; nickel ; the nickel - chromium - aluminium alloy 'Alnicro'; the nickel-chromium alloy 'Nichrome V'; the complex nickel-chromium-base alloys 'Inconel' and 'Inconel X'; and two 18-8 unstabilized and stabilized chromium-nickel-molybdenum steels (A.I.S.I. Types 316 and 318), some specimens of which were

plated with chromium or various thicknesses of copper. Final tests were carried out on: 'Inconel X', which, in some specimens, was separated from the graphite by copper foil; 'Alnicro'; the unstabilized (Type 316) stainless steel, including specimens copper- or chromium-plated to various thicknesses. Hardness data and micrographs are presented.

Of the range of materials tested, copper showed the highest resistance to attack by carbon, while nickel was only slightly more reactive. Molybdenum formed carbides at the interface, but there were no signs of carbon penetration into the metal beyond the carbide layer. Of the alloys and steels studied, 'Inconel X' appeared to be the least affected when heated in contact with graphite. 'Alnicro' was less resistant than 'Inconel X', but more resistant than 'Nichrome V', 'Inconel' and Types 316 and 318 stainless steel. 'Inconel X' and 'Alnicro' are considered to hold promise for applications involving contact with graphite up to about 1850°F. (1008°C.).

Copper, chromium and, to a much lesser extent, nickel, proved effective as barriers against diffusion of carbon into stainless steel, and copper or chromium electrodeposits on stainless steel were found to retard diffusion of carbon over long periods at temperatures up to about 1650°F. (900°C.). The effectiveness of copper as a diffusion barrier on 'Inconel X' was limited by interdiffusion of the copper with elements from the alloy: in this way the barrier was rendered ineffective at temperatures as low as 1650°F. (900°C.).

Erosion and Corrosion of Nickel-containing Materials in Wet Oxygenated Steam

H. A. CATALDI, C. F. CHENG and V. S. MUSICK: 'Investigation of Erosion and Corrosion of Turbine Materials in Wet Oxygenated Steam.'

Trans. Amer. Soc. Mechanical Engineers, 1958, vol. 80, Oct., pp. 1465–76; disc., pp. 1476–8.

Steam generated by boiling-water nuclear-power plants differs from that produced by conventional power stations in three respects: it is radioactive, wet, and contains free hydrogen and oxygen. Information on the behaviour of materials of construction exposed to such steam is extremely sparse. The literature suggests that the radioactivity of the steam would be of no great significance; few data are, however, available on the erosive effects of wet steam under high pressure, and little more on the influence of oxygen on the corrosion of metallic materials in the presence of such high-temperature water/steam mixtures.

The projected boiling-water reactor of the Dresden Nuclear Power Station of the Commonwealth Edison Company (U.S.A.) is designed to produce steam entering the turbine at a pressure of 869 p.s.i.a. (0·6 kg./mm.² absolute) and a temperature of 525° F. (274° C.). The steam will have an oxygen concentration higher than is normal in power-plant practice and a moisture content of ½ per cent. Selection of suitable materials of construction for the turbine components was therefore contingent on investigation of the resistance of potentially useful materials to corrosion and erosion by steam/oxygen/water mixtures under high-temperature and high-pressure conditions. The results of such an investigation are presented in this paper.

The materials studied are listed below. Compositions and details of heat-treatment are given in the paper.

Carbon Steels

Low-Alloy Steels

Molybdenum
Chromium
Chromium-molybdenum
Nickel-molybdenum

Nitrided Steels

'Nitralloy'
'EME' (19–12 chromium-nickel stabilized stainless steel)

Cast Irons

including 'Ni-Resist No. 3' (containing nickel 30, chromium 3, per cent.) and its ductile (spheroidal-graphite) counterpart

Stainless Steels

18–8 chromium-nickel austenitic steels (standard unstabilized, extra-low-carbon, and niobium-stabilized types)
High-chromium steels (three types)
12 per cent. chromium-molybdenum-tungsten-vanadium, chromium-molybdenum and chromium-niobium steels

Non-Ferrous Alloys

Cupro-nickels (90–10, 80–20 and 70–30)
'Monel'
'BTH' leaded bronze (copper 65, zinc 14, nickel 12, lead 5, tin 2·5, iron 0·76, per cent.)
'Stellite' 1 and 6 (complex cobalt-chromium-base alloys)

The *elastomer* 'KEL-F' was also included in the materials investigated.

The tests, which were devised to simulate conditions prevailing in high-pressure and intermediate turbines, were carried out in three phases:

(1) Erosion and corrosion tests using wet steam, at 350 p.s.i.a. (0·25 kg./mm.² absolute) and 430°F. (220°C.) max., containing 0·05, 80 and 110 p.p.m. of oxygen; pH 6·8.

(2) Erosion and corrosion tests with wet de-superheated steam, at 43 p.s.i.a. (0·03 kg./mm.² absolute) and 272°F. (133°C.), containing 70–110 p.p.m. of oxygen; pH 8·8–9·5.

(3) Pitting-, crevice- and galvanic-corrosion tests, using autoclaves, in water and steam at a pressure of 1015 p.s.i.a. (0·7 kg./mm.² absolute), a temperature of 545°F. (285°C.), and oxygen contents of 50 and 100 p.p.m. In one series of tests the specimens were exposed alternately to steam and water; in other tests specimens were exposed to condensing

steam or immersed in water. Only static corrosion tests were made in this phase of the investigation.

Resistance to erosion was assessed by its effects as manifested in the form of surface or washing erosion, 'wire-drawing' or channel erosion, and impact erosion, and was measured by weight loss and depth of pitting. Corrosion rates are presented in mils per year.

From the extensive data obtained the authors draw conclusions which they summarize as follows:

'(1) On the basis of the results of this preliminary evaluation, erosion and corrosion of a turbine in a boiling-water nuclear-power system can be maintained within tolerable limits by proper design and selection of special materials for critical areas. This evaluation is being supplemented by continuing laboratory tests, and by the operation of a 5000-kw. turbine directly connected to a 10,000 kw. boiling-water reactor at the Vallecitos Atomic Laboratory (Atomic Power Equipment Department).

'(2) In wet steam with low (0·05 p.p.m.) and high (100 p.p.m.) oxygen contents, stainless steels, 'Monel', and 'Stellite 6' were most resistant to 'wire-drawing' erosion. 'Ductile Ni-Resist 3', low-nickel steels, and low-chromium steels were better than low-molybdenum and carbon steels; plain cast iron was the least resistant of the materials tested.

'(3) High and low oxygen contents in wet steam, at pH 7 and 430°F., had no effect on the degree of erosion except for 'BTH' leaded bronze. With low oxygen concentrations, 'BTH' leaded bronze resembled low-chromium steel in erosion; with high oxygen, it resembled plain cast iron.

'(4) In wet steam at 300°–430°F. (150°–220°C.) and pH 7, both with low and high oxygen contents, stainless steel, 'Stellite No. 1', and 'Ductile Ni-Resist No. 3' were most resistant to corrosion. 'Monel' and low-alloy steels were superior to carbon steel. 'BTH' leaded bronze was the least resistant of the materials tested. At lower temperatures (300°–400°F.; 150°–205°C.) and low oxygen, the corrosion-resistance of the cupro-nickels was comparable to that of the most resistant materials; at high oxygen concentrations they very rapidly approached the extreme corrosion rate of 'BTH' leaded bronze.

'(5) At 300°–430°F., pH 7, an increase in the oxygen level from low to high increased the corrosion rate of all the materials tested except stainless steel. In the case of carbon steel, this effect of oxygen on corrosion diminished rapidly with increasing temperature. With the cupro-nickels, the effect of oxygen was much more severe at 400°F. than at 300°F.

'(6) Corrosion rates of all the materials exposed, at 300°–430°F., pH 7, to steam containing high and low concentrations of oxygen, increased with temperature, the logarithm of the corrosion rates varying approximately linearly with the reciprocal of the absolute temperature.

'(7) At 430°F., pH 7, in steam with high oxygen concentrations, no intergranular corrosion was found in standard and low-carbon grades of sensitized stainless steel in 1000 hours, nor were there any cracks around heavily stamped crosses.

'(8) With low oxygen contents, at 300°–430°F., pH 7, no pitting was observed except in the case of carbon steel, where the effect was minor. With high oxygen concentrations, all the materials pitted, especially at crevices. Stainless steel and 'Stellite No. 1' were the most resistant. 'Monel', cupro-nickel, and 'BTH' leaded bronze were better in this respect than low-alloy steel. Severest pitting was found in carbon steel.

'(9) In autoclave tests in steam/water/oxygen mixtures at 545°F., 1015 p.s.i.a., and containing 50 and 100 p.p.m. of oxygen, run for 6 and 10 weeks, the rates of corrosion decreased with time. The maximum depth of pitting, however, did not increase significantly.

'(10) In the autoclave tests, the order of resistance, as measured by the maximum depth of pitting, was the same as that for resistance to general corrosion. There was less difference in the maximum pitting depths among the ferrous materials tested than in the corresponding weight losses. This reflects the greater number of pits in the least resistant alloys.

'(11) There were no significant galvanic or crevice effects in the autoclave tests. Naturally-formed deposits of iron oxides were more effective in promoting pitting in all classes of steel tested than were artificial 0·002-in. (0·05-mm.) crevices.

'(12) Cast iron and low-carbon cast steel were subject to occasional very deep pits in the autoclave tests. These may be related to inclusions and/or pin-hole porosity.'

Susceptibility of Austenitic Steels to Intergranular Corrosion not associated with Carbide Precipitation

C. EDELEANU: 'The Intercrystalline Corrosion of Stainless Steels.'

Chemistry and Industry, 1958, No. 42, Oct. 18, pp. 1360–1.

Intercrystalline corrosion of austenitic steels is generally assumed to be the result of carbide precipitation, which, in practice, may be successfully controlled either by heat-treatment or by stabilization with titanium or niobium. The literature, however, indicates that there are at least three types of intercrystalline corrosion associated with austenitic stainless steel which cannot be attributed to lack of stabilization, and cannot therefore be prevented merely by ensuring that, on the basis of the conventional copper-sulphate or nitric-acid tests, the steel is not susceptible. The author discusses the three typical failures in this short article.

Failure of stabilized austenitic steel by intercrystalline corrosion in caustic solutions at elevated temperatures was described by WANKLYN and JONES (see abstract in *Nickel Bulletin*, 1958, vol. 31, No. 11, p. 302).

Intercrystalline corrosion was also reported, in 'Stress-Corrosion Cracking and Embrittlement' (CHAPMAN AND HALL, LTD., London, 1956; see *Nickel Bulletin*, 1957, vol. 30, No. 6, p. 100), to have occurred in low-carbon 18-8-molybdenum steels exposed to concentrated chloride solutions. Susceptibility to this type of failure was particularly pronounced in steels which had been solution-treated at a rather high temperature (e.g., 1200°C.) and in steels which had proved resistant to intercrystalline attack by copper sulphate or nitric acid. The author suggests, as a possible mechanism for this type of attack, the increased formation of delta phase at the grain boundaries at temperatures above 1050°C. On cooling, a great deal of the delta phase is transformed to austenite poor in nickel and rich in chromium. Since it is known that a low-nickel austenite is prone to cracking in chloride salts, it might well be that cracking follows the low-nickel regions of the metal, to give the general appearance of intercrystalline cracking.

Finally, intercrystalline corrosion has been observed in austenitic steels exposed to fused chloride. This type of attack is believed to be due to preferential leaching of the chromium by the salt, which, because of the higher diffusion rates at the grain boundaries, produces cracking resembling that of intercrystalline corrosion.

Stress Corrosion of Chromium-Nickel Stainless Steel in High-Temperature Lithium Hydroxide

G. E. GALONIAN: 'Results of Stress-Corrosion Tests of Type 347 Stainless Steel in 500°F. (260°C.), pH 10-11 Lithium Hydroxide.'

U.S. Atomic Energy Commission, General Electric Co. Report KAPL-M-GEG-11, May 2, 1958; 15 pp. + figures and tables.

Corrosion of materials of construction used in high-temperature water systems may be minimized, and the amount of transportable corrosion products decreased, by maintaining the pH of the primary coolant water at a high level. In this connexion lithium hydroxide is currently being investigated as an additive to raise the pH to the range 9·5-10·5. Use of such a dilute hydroxide solution would, under normal conditions, involve no corrosion problems. In some areas of the primary system, however, the possibility exists of accelerated corrosion or stress-corrosion cracking of austenitic stainless steel, due to concentration of the lithium hydroxide, mainly as a result of boiling in heated crevices or build-up of deposits by leakage to the atmosphere.

To study the effects of these conditions, two series of stress-corrosion tests were made on niobium-stabilized 18-8 chromium-nickel stainless steel. To evaluate the effects of solution concentration at heated crevices, tests were made on three threaded bolt assemblies, heated to 626°, 665° and 694°F. (330°, 352° and 368°C.) to induce boiling of the solution, and exposed for four weeks. Corrosion resulting from concentration by leakage was evaluated, by means of a Schroeder embrittlement detector, on a stressed specimen subjected to a small leakage of water from the system to the atmosphere. The temperature of the loop water was maintained at approximately 500°F. (260°C.), and the pressure at 2000 p.s.i.g. (1·4 kg./mm.^{2} above atmospheric pressure).

The corrosion scales found on specimens subjected to the boiling tests were thicker than expected, and metal penetration of about 0·001 in. (0·025 mm.) occurred in some areas of the threads of bolt specimens. No sign of stress-corrosion cracking was found. Deposits of calcium carbonate were present, but none of lithium hydroxide. After four weeks' exposure to the leakage tests, a mixture of calcium carbonate with some lithium compounds had formed on the surface of the specimens, but no severe corrosive attack was observed.

It is concluded that, under test conditions involving exposure to boiling solutions, Type 347 stainless steel does not appear to be susceptible to cracking, though accelerated corrosion may occur in crevice areas. Similarly the results are taken to indicate that exposure to conditions simulating leakage of the lithium-hydroxide solution to the atmosphere does not cause accelerated corrosive attack.

Cathodic Protection of Stainless Steels by Applied E.M.F.

J. G. HINES and T. P. HOAR: 'Stress-Corrosion Cracking of Austenitic Stainless Steels with Applied E.M.F.' *Jnl. Applied Chemistry*, 1958, vol. 8, Nov., pp. 764-76.

Although many stress-corrosion-cracking processes are at least partly electrochemical, there has been but little study of the effects, on the progress of such cracking, of the application of e.m.f. leading to external current. This paper reports an investigation of the influence of small cathodic and anodic polarizations, on the stress-corrosion cracking of austenitic stainless steels in hot aqueous magnesium-chloride solutions. Particular attention is devoted to the possible use of cathodic protection as a means of preventing or delaying onset of cracking.

In earlier work the authors have shown that the overall stress-corrosion-cracking process in such materials in hot concentrated chloride solutions may be divided into an induction period and a period of crack propagation. During the first (induction) stage the corrosion processes are very similar to those occurring in unstressed metal of similar type, and very little loss of strength occurs until the crack-propagation stage is entered. Since the presence of even small cracks is undesirable in a metal structure such as a chemical plant, the possibility of extending the induction period by cathodic protection would be of considerable practical interest.

The material used in the investigations now reported was 0·02-in. (0·5-mm.) diameter wire of an austenitic steel containing chromium 17·1, nickel 9·2, carbon

0·08, per cent., and a few confirmatory experiments were made on steels of the 18-8-Ti, 18-8-Nb, and 18-10-3Mo-Ti types. All were materials which had been used in earlier sections of the authors' extensive researches on stress-corrosion cracking of austenitic steels. Specimens were tested in the 'as-received' condition (fully softened but slightly cold-worked in coiling and uncoiling), after swabbing successively with carbon tetrachloride and acetone. An applied stress of 48,000 p.s.i. (21·5 t.s.i.; 38 kg./mm.^{2}) was used, selected as being near to the 0·1 per cent. proof stress of the steel. The corrosive environment was an aqueous solution of magnesium chloride containing 42 wt. per cent. of $MgCl_2$, prepared from pure crystals of the hexahydrate. The solution was maintained at its boiling point (154°C.) during the whole of the runs, and a slow stream of air was blown through it. A 6·5 cm. length of wire was exposed to corrosive attack. The behaviour of the steels was observed under conditions of cathodic or anodic treatment of the metal during the induction period prior to crack initiation and/or during crack propagation.

From the results, which are reported *in extenso*, the authors draw the following conclusions:

Under the experimental conditions used, initiation of stress-corrosion cracking in plain 18-8 chromium-nickel steel (which normally occurs after about 2 hours) is delayed for more than 200 hours by cathodic polarization producing an external current density of 50 μa./cm.^{2} or more. At lower polarizations crack initiation may be delayed for up to 60 hours. During prolonged cathodic treatment a substantial film containing magnesium hydroxide may be deposited as a result of the accumulation of cathodically produced hydroxyl ion; once this film has been formed, polarization giving a current density of 15 μa./cm^2. is sufficient to maintain protection. Galvanic cathodic protection may be attained by coupling with copper, mild steel and probably other metals and alloys.

Anodic treatment at current densities of 50 μa./cm.^{2} or even less hastens initiation of stress-corrosion cracking.

The propagation of stress-corrosion cracks is arrested immediately and permanently by cathodic polarization that reduces the potential to about −0·14 v (N-hydrogen scale); this produced an external current density of 80–170 μa./cm.^{2} under the conditions recorded. Smaller polarizations reduce the rate of propagation, which may fall to zero with passage of time.

Although mild anodic treatment does not accelerate the rate of crack propagation, it increases the number of cracks formed.

The results of the experiments are in harmony with the hypothesis of crack propagation by yield-assisted anodic dissolution of the advancing edge of a crack at current densities of approx. 1·5 a./cm.^{2}, this high current-density being maintained by continual yielding just ahead of the crack, which assists the egress of metal cations from the lattice and also prevents the formation of passivating films.

The observations made indicate that cathodic protection is a promising method of preventing stress-corrosion cracking in practice, particularly if protection can be applied locally to regions where the liability to cracking is high. While it is often possible to fabricate large portions of a plant from fully-softened material without introducing either cold work or residual stress, and also in favourable cases to provide stress-relieving treatments after fabrication, it can rarely happen that the final structure consists everywhere of completely unstressed, perfectly softened material. Development of reliable protective methods, even if only locally applicable, would therefore be practically useful. The authors make the following comments as to the advantages and limitations of this method of protection: 'It is unlikely that complete cathodic protection over the entire working surface of a chemical plant could be maintained; not only might the external current produced by a sufficient polarization be large, but the 'throw' into long narrow passages such as tubes would be poor. However, *local* protection of regions known to contain residual stress and/or to have been cold worked, or with a history of liability to stress-corrosion cracking, may well be practical in many cases. Galvanic anodes—copper (in concentrated chloride solutions) or mild steel—would offer the usual advantages of eliminating the need for an external source of e.m.f., insulated leads and reliable insoluble anodes, and could be built into appropriate regions of the structure or even provided as sprayed coatings or built-up metal in suitable cases. On the other hand, they would have the usual disadvantages of uncertain life and of lack of means of control; moreover, they would produce some contamination of the electrolyte, which, although unimportant in saline cooling waters, would be undesirable in process liquors. The dissolution of copper and its re-deposition as oxide (referred to in the paper) would introduce further complications, which might be detrimental or beneficial; copper anodes for any particular problem would evidently need careful preliminary investigation. An external source of e.m.f. and 'insoluble' anodes such as carbon or platinum would pose other problems of design, but, provided that the anodes were indeed insoluble (platinum dissolves anodically in hot concentrated chloride solutions), might offer the most reliable means of practical cathodic protection for stainless-steel chemical plant.'

Resistance of Nickel-containing Materials to Corrosion by Liquid Sodium

R. F. DUDEK and K. M. FERGUSON: 'The Corrosion Testing of Various Materials in Sodium.'
Babcock and Wilcox Co., Report No. 7020, Apr. 26, 1957; 35 pp.

The work reported forms part of a test programme carried out with the aim of accumulating data on the corrosion-resistance of various materials of construction to liquid sodium at 1100°F. (595°C.).

MARKERT, in a previous report (*Babcock and Wilcox Co., Report No.* 3792), had given the results of a series of 500-hour corrosion tests on certain materials, which, however, did not include all those of interest to Atomic Power Development Associates, the organization concerned. Further tests were therefore carried out to obtain information on the behaviour of the following groups of materials when exposed to liquid sodium: carbon steel; standard, molybdenum-containing, and stabilized 18-8 chromium-nickel stainless steels (Types 304, 316 and 347); 'Croloy' low-alloy steels (containing ½, 1¼ and 2¼ per cent. of chromium respectively); 'A' nickel; zirconium; 'Inconel' and 'Inconel X'; straight-chromium stainless steels; and carbon steel clad with 18-8 stainless steel.

The specimens were exposed for 1000 hours, using a modified enclosed-rotor pump for the corrosion loop. Tests were made both with static and revolving specimens. The oxygen content of the sodium was considered to be of importance and efforts were made to measure and control the amounts present. In three of the four tests this element was controlled by means of a diffusion-type cold trap; in the fourth a flow-through cold trap was used. Oxygen content was measured chemically and by use of a plugging indicator.

The report is presented in two sections: the first records and discusses the corrosion rates of the materials; the second gives the results of metallographic examination of the specimens after exposure.

The main results and conclusions are summarized as follows:

'(1) Carbon steels and the Croloy steels decarburized almost completely as a result of the exposure to sodium at 1100°F. (595°C.) Weight loss for those materials could be largely attributed to loss in carbon. No other attack was noted.

'(2) Types 304, 347 and 316 stainless steels carburized when any carbon was available. They were not attacked by sodium, regardless of structural condition.

'(3) 'A' nickel was superficially attacked. It appears that this attack is a leaching-out process of some of the nickel by sodium. The weight-change data were irregular.

'(4) 'Inconel' and 'Inconel X' revealed no structural changes or selective corrosion, but a weight loss in both materials indicates that general-solution type of attack probably occurred.

'(5) Zirconium developed a hard and brittle surface layer of limited depth, apparently due to oxygen absorption. A weight gain was noted.

'(6) A change in surface composition in the form of a film less than 1 micron in thickness was noted on some of the specimens examined by X-ray methods. This examination indicated that metal may have been transported in the system.'

Resistance of Nickel-containing Materials to Hydrofluoric Acid

H. A. PRAY, F. W. FINK, B. E. FRIEDL and W. J. BRAUN: 'Progress Report on Corrosion-Resistant Materials for Hydrofluoric Acid.'

Battelle Memorial Inst., Report 268(*Del.*), June 15, 1953; 70 pp. (Recently declassified.)

Among the many corrosion problems arising in the production of uranium tetrafluoride and in the extraction of uranium metal from ores and concentrates, some of the most serious are those occurring in plant handling hydrofluoric acid. Corrosion in this acid frequently results in evolution of hydrogen, and the fluoride ion released may either form protective fluorides (as is the case with magnesium and, to a lesser extent, with iron) or, if oxygen is present, the ion may form, with some metals (e.g., copper), oxides which are much more soluble than the pure metal.

The first phase of the investigation, reported earlier, showed that the following materials could be considered for plant handling hydrofluoric acid: platinum, silver, 'Monel', cupro-nickel, copper, lead and steel. This subsequent report discusses some of the other characteristics of these materials and gives details of corrosion studies made under conditions simulating those which could occur in the type of plant under consideration. From the large amount of experimental data recorded, the following conclusions are drawn.

'Platinum is unaffected by boiling hydrofluoric acid (38 and 48 per cent.) or by hydrofluoric acid (50–70 per cent.) at 140°F. (60°C.). Addition of oxygen to the system does not promote attack.

'Silver also shows excellent resistance to the above conditions, although a measurable rate of attack is observed. When oxygen is added to the acid, slightly increased corrosion occurs, but the additional attack is not sufficient to change the rating of silver as excellent. Sterling silver shows corrosion-resistance approaching that of pure silver.

' 'Monel' is resistant to 50, 65 and 70, per cent. hydrofluoric acid at 140°F. (60°C.), provided that oxygen is eliminated from the vapour phase. With oxygen present the rate of attack is markedly increased. At any given oxygen level, 'Monel' is much less resistant to 50 per cent. hydrofluoric acid than to 65 or 70 per cent. acid. If oxygen in the gas - purging mixture is restricted to less than 1500 p.p.m. in the vapour phase, 'Monel' has proved very resistant to boiling chemically-pure 38 and 48 per cent. acids, but at that oxygen level corrosion-resistance in commercial grades of acid of the same concentration varies.

'With oxygen concentrations of 2000 p.p.m. or less, cupro-nickel alloys showed low rates of attack: beyond that concentration of oxygen corrosion rates suddenly increased.

'Addition of various other elements to cupro-nickel were made to determine whether complex alloys of this type would exhibit better corrosion-resistance than straight cupro-nickel, but a phosphorus addition was the only one which conferred any benefit.

'Lead and several lead alloys gave poor resistance to boiling hydrofluoric acid.

'Steel showed good resistance to 70 per cent. acid at 90° and 140°F. (32° and 60°C.) in both the liquid and the vapour phases, and, unlike copper and copper alloys, was not markedly affected by the presence of oxygen in the corroding system.'

ANALYSIS

At the request of some of our readers who are particularly interested in methods of analysis, we are including, in this and future issues of *The Nickel Bulletin*, a section in which reference will be made to items concerned with such techniques.

Colorimetric Determination of Nickel
See p. 6.

Determination of Nickel by Solvent Extraction
See p. 6.

Gravimetric Separation of Copper, Nickel and Zinc
See p. 6.

PATENTS

Extraction of Nickel from Silicate Ores

The two patents cover processes for extraction of sulphur-free high-purity nickel from silicate-containing ores such as the garnierite deposits of New Caledonia.

The treatment according to *Pat.* 1,160,093 involves mixing the ground ore with powdered carbon, sodium chloride and calcium carbonate, and roasting in a reducing atmosphere at a temperature above 750°C. The nickel is then dissolved, in the form of nickel ammine, by treatment of the reduced ore at 30°–50°C. with an ammoniacal solution containing an ammonium salt (preferably ammonium carbonate). The nickel may be finally extracted by an evaporation/concentration operation and electrolysis of the treated solution, or, in the case of an ammoniacal ammonium-carbonate solution, by precipitation and oxidation of the nickel carbonate, and reduction of the nickel oxide.

The procedure covered by *Pat.* 1,160,094 comprises the following stages: (1) The nickel content of the roasted ore is rendered soluble by transformation to the chloride by contact, at a temperature of 180°–500°C., with a gaseous mixture of hydrochloric acid and reducing agents; (2) the chlorides are extracted by treatment with a solution of HCl of a concentration less than 10 per cent.; (3) final extraction of nickel may be effected by electrolysis or by hydrolysis and precipitation treatment.

REGIE NATIONALE DES USINES RENAULT.
French Pats. 1,160,093, 1,160,094.

Continuous Production of Supported Free-Metal Catalysts by Decomposition of Nickel Carbonyl

Supported metal-film catalysts suitable for use in processes catalyzed by the presence of free metal are produced by thermal decomposition of a metal carbonyl (preferably of nickel, cobalt, iron, molybdenum, tungsten or chromium) in contact with a fluidized support material (preferably silica gel, activated alumina or silica-alumina, separately or in combination). The metal-coated support material is then passed into the hydrogenation reactor, and, after use, the spent supported catalyst is treated with carbon monoxide to re-form the metal carbonyl and recover the support material, preparatory to re-circulation in the catalyst-preparation zone. A flow sheet illustrates the stages of the cycle.

The preferred application, hydrogenation of benzene to cyclohexane, involves use of activated alumina as the support material and decomposition of nickel carbonyl to obtain the free metal. Details of procedures and operating conditions for nickel, cobalt, molybdenum and tungsten carbonyls are given. Advantages claimed are: increased catalytic activity, rapid preparation and immediate use of the catalyst, and the possibility of continuous re-cycling.

E. L. MILLER, assignor to PURE OIL CO.
U.S. Pat. 2,846,488.

Nickel-containing Dehydrogenation Catalyst

Catalysts particularly suitable for production of carbonyl compounds from alcohols consist of copper, nickel, and one or more alkali-metal compounds, on a siliceous support, the content of alkali-metal compound being 1–30 per cent. of the combined weight of the copper and nickel. Processes for the production of the catalyst and dehydrogenation of an alcohol are also covered. The preferred application is to production of an aliphatic ketone from a secondary alcohol.

RHEINPREUSSEN AKTIENGESELLSCHAFT FÜR BERGBAU UND CHEMIE. *Brit. Pat.* 803,373.

Calcium/Nickel-Phosphate Dehydrogenation Catalyst

Dehydrogenation of a monoolefin having at least four carbon atoms in the olefin chain is effected by contact with a calcium/nickel-phosphate catalyst (preferably a calcium/nickel-phosphate/chromium-oxide catalyst). The preferred application is to dehydrogenation of *n*-butylene to butadiene-1,3.

POLYMER CORPN., LTD. *Brit. Pat.* 803,459.
Similar to *Belgian* „ 557,369.

Bright-Nickel-Plating Solutions

Bright, ductile nickel deposits are produced from an aqueous acidic solution containing one or more of the salts nickel sulphate, chloride, fluoborate and sulphamate, approx. 0·1 g./L. to saturation of at least one organic sulphon compound (the appropriate classes of which are listed), and about 0·002–0·5 g./L. of a compound selected from the following: vinyl

substituted m-dioxane, and di-vinyl substituted bi(m-dioxane), and their derivatives in which the m-dioxane ring is substituted with a chloro, bromo, hydroxy, methoxy, ethoxy, and hydroxy-ethoxy radical. Preferred organic sulphon compounds are allyl sulphonate and 2-butyne-1,4-disulphonate; 3,9-divinyl spirobi (m-dioxane) and 3-vinyl m-dioxane are covered as vinyl substituted m-dioxane additions.

D. H. BECKING and H. BROWN, assignors to UDYLITE RESEARCH CORPN. *U.S. Pat.* 2,852,449.

Deposition of Hard, Ductile, Compressively-Stressed Dull-Nickel Coatings

The coatings are deposited from an aqueous nickel-plating solution containing a nickel salt (preferably nickel sulphate and/or chloride, nickel sulphamate and/or chloride, or a combination of nickel sulphate, sulphamate and chloride) together with an ortho-formylbenzenesulphonic acid or its salts (preferably the sodium salt) having not more than two sulphonate groups in the benzene ring. The reagent is added in amounts less than 1 per cent. of the solids present in the electrolyte and sufficient to obtain, at an operational temperature in the region of 130°F. (55°C.) and at current densities within the range 35–140 amp./sq. ft. (3·7–15·1 amp./dm.^{2}), a dull-nickel deposit in which the residual stress is compressive, the Vickers hardness ranges from 440 to 710, and the ductility is such that the deposits withstand without cracking a hammer blow sufficient to dent the basis metal. Examples of suitable solutions and operating conditions are given.

The hardness, ductility and compressive stress of such coatings render them ideal for applications where impact-resistance and high adhesion are required, e.g., aeroplane propeller blades.

D. S. CARR, assignor to BART LABORATORIES CO., INC. *U.S. Pat.* 2,842,487.

Deposition of Nickel on Dielectric Surfaces

Deposition of nickel/nickel phosphide on dielectric surfaces is achieved by (1) sensitizing the surface by vacuum deposition of a thin film of metal (e.g., gold, copper, iron or nickel, but preferably aluminium) which will serve, directly or indirectly, as a catalyst in the subsequent plating process; (2) chemical deposition of a coating of nickel/nickel phosphide from a hot aqueous solution of a nickel salt and a hypophosphite. Sensitization may be preceded by a surface-cleaning process involving ionic or electron bombardment.

The invention is claimed to be of particular advantage for deposition of nickel/nickel phosphide on polytetrafluoroethylene and dielectric films too thin to undergo the surface roughening necessary prior to conventional sensitizing processes. Application of the process to capacitors and printed circuits is covered. Preferred plating solutions are given.

BRITISH DIELECTRIC RESEARCH, LTD. (inventors, D. S. MARGOLIS and T. E. SHEN). *Brit. Pat.* 802,053.

Bonding of Heat-Exchanger Components by Electroless Deposition of Nickel-Phosphorus Alloy

The components are brought into intimate contact, preferably under pressure, and are treated in an aqueous plating solution containing a nickel salt, a hypophosphite, glycollic acid and sodium hydroxide, with resultant deposition of an alloy containing 85–97½ per cent. of nickel, and 2½–15 per cent. of phosphorus. The parts are treated sufficiently long to form an adherent connecting web or fillet of deposited alloy between the portions to be joined. The patent also covers the subsequent heating of the plating-bonded parts at a temperature of about 650°F. (345°C.), i.e., below the melting point of the deposited metal, and thereafter heating the parts to a temperature at least as high as the melting point of the material, to melt and spread it and thus effect brazing, thereby utilizing the deposited nickel-phosphorus alloy as brazing material.

CHRYSLER CORPN. *Brit. Pat.* 804,334

Nickel Plating of Aggregates viâ the Carbonyl

Aggregates produced by compacting and sintering such hard materials as carbides and silicides contain small interstices which affect the properties of the material, particularly the electrical characteristics. According to this invention, such interstices are filled with metal, thereby improving physical, electrical and mechanical properties of the aggregate: for example, the electrical conductivity of silicon carbide, as used for electrical furnaces, may be increased in this way. The compacted aggregate is heated to a temperature above that of thermal decomposition of a gaseous metal compound, and the surface is then cooled (for example, by contact with CO_2) to below the decomposition point of the compound. The metal-bearing gas is permitted to permeate the aggregate, and deposition of metal occurs within the internal interstices. The temperature within the aggregate is maintained in part by the exothermic reaction itself and in part by induction heating. As more gas is introduced deposition of metal proceeds progressively from the interior of the aggregate towards the surface, until the interstices are completely filled. Introduction of a softer metal prior to sintering of hard metal powders would improve the adherence of the particles. Details are also given of the technique and apparatus used to gas-plate metal powders prior to compacting.

Five decomposable compounds are claimed for use within the scope of the patent: nickel carbonyl, copper acetylacetonate, chromium hexacarbonyl, tungsten carbonyl and molybdenum carbonyl. Gas plating of silicon-carbide aggregates viâ nickel carbonyl is described.

P. R. MARVIN, assignor to COMMONWEALTH ENGINEERING CO. OF OHIO. *U.S. Pat.* 2,847,319.

Gas Plating of Heat-Sensitive Fibres

Vegetable, semi-synthetic or synthetic fibres sensitive to heat, produced by extrusion of a thermoplastic

resin, are metallized, without loss of textile characteristics, by first coating the fibre with a heat-resistant silicone resin which then provides a protective surface for deposition, by thermal decomposition, of the metallic constituent of a heat-decomposable gaseous compound (metal carbonyls, hydrides, alkyls, halides, nitroxyls or nitrosyl carbonyls). Metallization is accomplished preferably during spinning of the fibre: the heat required to soften and extrude the thermoplastic material is sufficient to facilitate application of the heat-resistant coating and decomposition of the gaseous compound. *Inter alia*, carbonyls of nickel, iron, chromium, molybdenum and cobalt are recommended for use as decomposable gases.

F. E. DRUMMOND, assignor to COMMONWEALTH ENGINEERING CO. OF OHIO. *U. S. Pat.* 2,847,322.

Application of Adherent Sprayed Coatings of Nickel-containing Materials

The invention relates to the production of strong, adherent coatings of heat- and/or corrosion-resisting materials such as stainless steel and nickel- or cobalt-base alloys. The method, according to the invention, is designed to secure improved diffusion across the interface between the sprayed coating and the base, and across the interface of separate particles within the layer. The stages of the process are as follows:

(1) Application, by spraying, of a coating, preferably 0·001–0·002-in. (0·025–0·05 mm.) thick, consisting of one or more metal borides and/or an alloy containing not less than 2 (preferably 2–8·5) per cent. of boron: ferro-boron or nickel-boron are suitable alloys. This interlayer is stable at the spraying temperature, but sufficiently unstable at higher temperatures to cause the boron compounds to break down, or otherwise to release the boron present.

(2) Application, by spraying, of the main coating, which should preferably contain 0·02–0·08 per cent. of boron.

(3) Heating, in a reducing or non-oxidizing atmosphere, at a temperature sufficient to cause the boron content of the interlayer to diffuse into, and bond together, the basis metal and the main coating, but not high enough to have any deleterious effects on either component or coating.

By way of example, details are given of procedures used to spray coatings of 'G 42B' stainless steel and 'Nimonic 90' viâ an interlayer of the boron-containing alloy 'Colmonoy No. 6'.

BIRMINGHAM SMALL ARMS CO., LTD. (inventor, E. H. CARMAN). *Brit. Pat.* 803,649.

Rubber-Adherent Tire-Bead Reinforcing Wire Resistant to Atmospheric Corrosion

Attempts to render ferrous tire-bead reinforcing wire corrosion-resistant by coating with galvanized zinc, followed by a coating of copper to confer the adherence to rubber necessary for vulcanization, have proved unsatisfactory under certain conditions: in particular, the treated wires tend to lose their adherence to rubber after long storage.

The patent covers a process which, though of general applicability, is peculiarly suited to provision of tire-bead wires which will maintain their adhesive characteristics under long periods of storage or severe atmospheric conditions. The wire is first coated with a layer of hot galvanized zinc to a thickness of at least 0·000002 in., upon which is electrodeposited a thin layer of nickel. This intermediate nickel coating, which has been found to achieve an extraordinary adherence over a longer period, is finally electroplated with copper to a thickness which will permit satisfactory vulcanization. Copper- and nickel-plating solutions and procedures are described.

NATIONAL-STANDARD CO. (inventor, O. E. ADLER). *Brit. Pat.* 801,298.

Production of Nickel-Plated Aluminium Propeller Blades

Hard protective coatings of nickel having improved adherence are obtained on aluminium or aluminium-alloy aircraft blades by interposing sprayed laminated layers of rubber-like materials of specified types, finishing with a non-metallic layer of a rubber-like adhesive containing an electro-conductive carbon black in colloidal dispersion, suitable as a base for electrodeposition of nickel. Details are given of thicknesses of the respective laminations and of intermediate baking treatments necessary to cure the adhesives.

UNITED AIRCRAFT CORPN. *Brit. Pat.* 804,287.

Copper-Nickel-Silicon Alloy Resistant to Stress Corrosion

Alloys claimed to be highly resistant to stress corrosion, season cracking, and oxidation, and to have high tensile strength, fall within the following composition range: aluminium 3–10, nickel 3·5–5, iron 3·5–5, silicon 0·7–2, per cent., remainder copper. The iron content is stated to be critical: alloys containing more than 5 per cent. of iron have been shown to be susceptible to failure by stress corrosion and to have inferior machining and hot- and cold-working characteristics.

GENERAL ELECTRIC CO. *Brit. Pat.* 802,044.

Soft Soldering of Ceramic Components

Soft soldering of non-metallic refractory bodies to metallic or non-metallic materials is facilitated by coating the non-metallic refractory with a mixture comprising about 60–94·5 wt. per cent. of a heavy metal (nickel, cobalt, iron), preferably in powder form, about 0·5–30 wt. per cent. of a manganese constituent (calculated as MnO_2) and about 30 wt. per cent. of powdered glass. The patent describes methods of preparing the coating, which may be applied in any convenient manner (dipping, brushing on, etc.) as a suspension, in a liquid vehicle, of the ingredients named. The coated body

is then fired at temperatures of 1300°–2400°F. (705°–1315°C.) for 15 minutes–2 hours, in a reducing atmosphere.

The coated material is then ready for soft soldering to a metallic or metal-coated body.

D. W. LUKS, assignor to FRENCHTOWN PORCELAIN CO. *U.S. Pat.* 2,848,802.

Nickel-(Cobalt)-Molybdenum-Aluminium Alloy for Battery-Valve Cathodes

Cathodes for use in battery valves consist of an alkaline-earth emissive coating and a filamentary core made from an alloy containing molybdenum 10–40 (preferably 20), aluminium (as a reducing agent) 0·1–2 (preferably 0·8–1·2) per cent., remainder nickel and/or cobalt. Compared with alloys currently used for the same purpose, an alloy of the preferred composition is claimed to have greatly increased hot strength, less tendency to recrystallization and higher resistivity. Emission properties are claimed to be at least as good after 500 hours' operation as those of cathodes made from conventional nickel alloys, and are very much better than those of cathodes in which the core is of tungsten.

MULLARD RADIO VALVE CO., LTD. (inventors, C. H. R. GENTRY and G. L. DAVIS). *Brit. Pat.* 801,185.

Nickel-Manganese Alloy for Cathode Cores of Thermionic Valves

The alloy contains nickel 98, manganese 0·2–2, per cent. The manganese addition is claimed to prevent separation of the oxide layer during operation of the thermionic valve, without deleterious effects on the emission characteristics of the cathode.

COMPAGNIE FRANCAISE THOMSON-HOUSTON (inventor, W. T. MILLIS). *French Pat.* 1,159,774.

Iron-Nickel-Aluminium Alloy for Permanent Magnets

The patent covers a casting alloy of the following composition: nickel 33·5, aluminium 12, silicon 0·7, titanium 0·35, per cent., remainder iron.

Y. S. SAKHATUNOV and E. G. PUCHNIN. *U.S.S.R. Pat.* 108,073.

Age-Hardenable Nickel-Chromium-Beryllium Alloy for Watch Springs

Thermally compensated balance springs for watches or clocks are fabricated from an alloy containing little or no iron, the temperature coefficient of elasticity of which is adjusted to a zero or near-zero value, over the required working temperature range of the spring, by control of the internal stress present in the material. The desired degree of internal stress may be achieved by the permanent superimposition of external tensile or compressive loads or external magnetic fields; by cold deformation; by ordering treatment; by selection of a suitable cooling rate after heating; by martensitic transformation; or by precipitation-hardening. The preferred alloy contains nickel 95, chromium 2·9, beryllium 2·1, per cent. The desired internal stress is achieved by subjecting the alloy to cold deformation in excess of 90 per cent. and age-hardening for 20 minutes at 500°C.

Springs so fabricated have greater corrosion-resistance and a higher degree of reproducibility and uniformity than the irreversible iron-nickel alloy used hitherto.

R. STRAUMANN, F. STRAUMANN and G. KRÜGER. *Brit. Pat.* 804,133.

Nickel Cladding of Molybdenum as an Aid to Brazing

Molybdenum or its alloys are sheathed with an alloy readily and uniformly wettable by brazing materials and having a coefficient of thermal expansion similar to that of molybdenum, e.g., an alloy containing cobalt 10–35, nickel 22–23, chromium 0–10, per cent., remainder iron. The composite is then heated to between 870° and 1320°C., and finally hot-rolled to achieve a section reduction of at least 30 per cent., thereby also achieving an intimate bond between the molybdenum and the sheath. An intermediate layer of palladium, copper, chromium or a nickel-copper alloy (containing 60–70 per cent. nickel) may be interpolated between the molybdenum and the sheath. The cladding facilitates the brazing of molybdenum either to itself or to other metals.

WESTINGHOUSE ELECTRIC CORPN. (inventors, E. F. LOSCO, J. H. BECHTOLD and J. P. WEBB). *Belgian Pat.* 561,983.

Nickel-Phosphorus Powdered Brazing Material

Brazing materials having properties superior to those of copper- or silver-base brazing alloys contain 85–97½ per cent. of nickel and 2½–15 per cent. of phosphorus, the nickel and phosphorus being present in powdered form either (1) as a mixture of nickel powder (e.g., carbonyl nickel) and red phosphorus powder, or (2) as a nickel-phosphorus powdered alloy produced by chemical reduction from a plating solution containing a nickel salt and hypophosphite, or (3) as a mixture of an alloy so obtained with nickel powder and/or red-phosphorus powder. The compositions covered have a melting point within the range 1650°–1800°F. (900°–980°C.) and a brazing temperature of between 1650° and 1800°F. (900° and 980°C.). They may be applied to the joint either in powder form or as a suspension in a suitable solvent. Details are given of plating solutions suitable for precipitation of alloys in the required composition range. The nickel-phosphorus alloys are intended mainly for use in brazing carbon and alloy steels: seven examples of brazing procedures are given. The following advantages are claimed for such alloys: reasonable cost; good wetting properties; use of brazing temperatures which avoid grain growth and distortion and oxidation of the parent metal; use of inexpensive commercial cracked-gas atmospheres; maintenance of strength in the stainless-steel parts; a nickel content which minimizes risk of oxidation of the joint at high temperatures.

CHRYSLER CORPN. *Brit. Pat.* 800,793.

High-Strength Nickel-Alloy Steels for Pressure Vessels

The steels covered are of firebox quality, suitable for forgings and for rolled plates, sheets, bars and rods. Together with a tensile strength in the range 100,000–125,000 p.s.i. (44·5–55 t.s.i.; 70·5–88 kg./mm.^{2}) the steels are claimed to possess satisfactory ductility and toughness, a high yield strength, improved weldability, and good impact properties down to −40°F. (−40°C.). Data on properties typical for the steels in the heat-treated condition are included. Three composition ranges are claimed (specifying, respectively, the general limits of the steels covered, and modifications optimum for steels up to, and over, a section thickness of 3 in. (7·5 cm.)). The type of steel is indicated by the general-purpose range shown in the table below.

J. G. ALTHOUSE, assignor to LUKENS STEEL CO. *U.S. Pat.* 2,853,379.

Gold-Nickel Thermocouple Elements

Two patents cover, respectively, alloys for the negative and the positive arms of thermocouples stated to give thermal e.m.f.'s per °C. higher than those hitherto attainable with high-melting-point ductile materials.

The negative element is made from a gold-nickel alloy containing 78–85·5 per cent. of gold, balance nickel. In the preferred composition the gold: nickel ratio is 7:5, giving a gold content of 82·5 per cent. The positive element consists of an iron-aluminium alloy which may also contain vanadium or molybdenum.

Both patents claim thermocouples in which the two types of alloy are used in the rôles indicated. Typical e.m.f. values are cited.

N. E. LINDENBLAD, assignor to RADIO CORPN. OF AMERICA. *U.S. Pats.* 2,846,493, 2,846,494.

Iron-Nickel-Chromium-base Age-Hardenable Turbine-Blading Alloys

Austenitic age-hardenable turbine-blading alloys claimed to combine high strength and corrosion-resistance at temperatures up to the region of 1200°F. (650°C.), with satisfactory weldability and ease of fabrication by forging and rolling, fall within the following limits of percentage composition: nickel 30–35, chromium 12–15, tungsten 5·5–7·5, molybdenum 2·5–5, titanium 1·5–3, aluminium 0·2–0·5, zirconium 0·01–0·5, carbon 0·04–0·1, Mn+Si+S+P not exceeding 0·2 per cent., remainder iron. The alloys are preferably cast *in vacuo*.

Data illustrative of mechanical properties exhibited by typical alloys subjected to various solution- and ageing-treatments are given.

GENERAL ELECTRIC CO. *Brit. Pat.* 800,989.
Similar to *Belgian* „ 557,872.

Chromium-Nickel Austenitic Steel for High-Temperature Applications

The steel is intended for applications involving resistance to mechanical stress at temperatures above 400°C. Consistently good properties are claimed to be obtained by means of a composition designed, by control of the silicon content and suitable deoxidation treatment, to eliminate the formation of deleterious low-melting silicates: the maximum silicon content is 0·3 (preferably 0·2) and the oxygen 0·008, per cent. Limits of composition of the other constituents are carbon 0·4 max., chromium 10–25, nickel 8–40, manganese 20 max., niobium+tantalum 4·5 max., titanium 3·0 max., per cent. Tungsten, molybdenum, vanadium or zirconium may be present up to a total percentage of 10, aluminium from 0·2 to 3·0 per cent., boron from 0·003 to 0·25 per cent., and nitrogen in amounts up to 0·3 per cent. Provision is made for the replacement of the nickel, in part or in toto, by cobalt.

DEUTSCHE EDELSTAHLWERKE A.G. (inventors K. BUNGARDT and H. SYCHROVSKY). *French Pat.* 1,165,303.

Nickel-base Austenitic Alloys for High-Temperature Applications

The alloys are intended for service involving resistance to mechanical stress at temperatures higher than 500°C. Reproducibility of properties is achieved by limiting the silicon and oxygen contents to a maximum of 0·3 (preferably 0·2) and 0·008 per cent., respectively. Major compositional requirements are: carbon 0·15 max., nickel 40–85, chromium 10–25, niobium+tantalum 4·5 max., titanium 3·5 max., per cent. The alloy may include manganese 3 max., aluminium 0·2–3·0, nitrogen 0·3 max., boron 0·003–0·25 per cent., and tungsten, molybdenum, vanadium or zirconium, separately or conjointly, up to a total content of 10 per cent. The patent provides for use of cobalt in place of nickel, with associated increase in the carbon content to 1 per cent. max.

DEUTSCHE EDELSTAHLWERKE A.G. (inventors, K. BUNGARDT and H. SYCHROVSKY). *French Pat.* 1,165,304.

Steels for Pressure Vessels

C %	Si %	Mn %	S %	P %	Ni %	V %	Mo %	Ti %	Cu %	Al %
0·08-0·30	0·15-0·35	0·90-1·40	0·050 max.	0·040 max.	1·25-1·75	0·05-0·13	0·10-0·30	0·005-0·05	0·35 max.	0·03-0·10

Non-Magnetic Precipitation-Hardenable Austenitic Chromium-Nickel Steels

Corrosion-resisting steels having good creep-resistance over a wide range of temperature, and capable of being age-hardened to achieve a yield strength of at least 35 tons/sq. in. (55 kg./mm.^{2}), fall within the following composition range: chromium 8–25, nickel $\geq$12, manganese 6–35, vanadium 0·5–4, silicon 0·2–2, carbon 0·37–0·9, per cent. Silicon and vanadium contents should be not less than 1 per cent. and the steel may contain also the following elements, in amounts not more than those indicated: molybdenum 5, tungsten 5, titanium 5, niobium and/or tantalum 5, aluminium 2, zirconium 2, beryllium 2, per cent.

Recommended heat-treatments and data on typical mechanical properties are given.

HADFIELDS, LTD. (inventors, J. R. RAIT, T. H. MIDDLEHAM and J. O. WARD). *Brit. Pat.* 803,816.
(Addition to „ „ 741,053)
Similar to *South African* „ 2046/56.

Production of Surface Cooling Ducts in Turbine Blades

The process involves channelling the surface of a powder-metallurgically-produced component, filling the channels with a temporary solid filling flush to the surface, and spraying the surface of the component with metal, to provide a heat- and corrosion-resisting sheath. The temporary filling is then removed from the enclosed channels, to leave ducts between the sheath and the core. Finally, adequate adhesion of the sprayed sheath to the basis metal is achieved by sintering. The main advantage claimed for the invention is that the sheath forms an adherent unitary seamless envelope of homogeneous metal which effectively encases the component, without involving the welding or brazing processes employed with sheaths of pre-formed material.

In an example illustrating the procedures used, 'Nimonic 90' powder is sprayed onto an initial coating of 'Colmonoy 6' (nickel-chromium-boron alloy), to provide the sheath for a 'Nimonic 90' turbine blade.

BIRMINGHAM SMALL ARMS CO., LTD. (inventor, D. A. OLIVER). *Brit. Pat.* 803,650.

Cobalt-containing Nickel-base Brazing Alloys

Nickel-base brazing alloys used to join stainless-steel cores to the skins of honeycomb-core structures have been found to diffuse, during the brazing process, into the ferritic stainless-steel foil, forming stable austenite. The difference in coefficients of expansion of the austenite and untransformed material results in a weak structure. Experiments with '17-7 P.H.' stainless steel have shown that addition of cobalt to the brazing alloy markedly reduces diffusion of the alloy, and that whatever solution of the '17-7 P.H.' foil does occur results in a structure similar to that of the parent '17-7 P.H.' material. The patent covers cobalt-containing brazing alloys within the following limits of composition: nickel 40–85, cobalt 5–50, chromium 0–20, boron 2–5, silicon 1–5, iron 0–15, carbon 0–0·5, per cent. Two preferred compositions are given below:

Ni %	Co %	B %	Si %	Cr %	Fe %	C %
70	20	3	4·5	—	—	<0·1
60	20	3	4·5	7	3	<0·1

The alloy is suitable also for brazing cobalt-containing basis metals, and may be used in the form of welding rods.

COAST METALS, INC. *Brit. Pat.* 803,253.

Gold-Nickel-base Alloys for Brazing Stainless-Steel Wire Cloth

The seams of interwoven stainless-steel wire cloth for fourdrinier wire and covers of cylinder moulds used in paper-making machines are fused together to form endless lengths by use of brazing alloys containing gold 82 or 75, nickel 25 or 18, per cent., or gold 72, nickel 22, chromium 6, per cent. Such seams are claimed to exhibit high resistance to the corrosive conditions involved in papermaking.

W. HAYDEN. *U.S. Pat.* 2,851,233

Production of Porous Nickel-containing Components

Components are produced from resin-coated metallic powders bonded together by partial sintering to form a porous skeleton, in the shape of the component, which is suitable for impregnation by a molten metal (preferably copper) the melting point of which is lower than that of the skeleton material. The skeleton is divided into at least two sections by the introduction of a barrier region containing particles of an addition metal (preferably silver) which is impervious to the infiltrant. Completion of the sintering process, while allowing the infiltrant to permeate the pores on one side of the barrier region, results in a finished product the porosity of which can be controlled to vary from 10 to 55 per cent., where and as desired. The invention is especially adapted for the production of porous bodies from stainless-steel, nickel and nickel-alloy powders. In discussion of the advantages resulting from the use of the process, it is mentioned that copper-impregnated parts of a stainless-steel article are, for example, more easily machined than the more porous regions.

J. P. SCANLAN and L. ALTER, assignors to SCHWARZKOPF DEVELOPMENT CORPN. *U.S. Pat.* 2,845,346.

'Monel' in Apparatus for Production of Uranium Fluoride

Uranium fluoride is produced by reaction between uranium oxide and gaseous hydrofluoric acid. The uranium oxide, in the form of pellets, is passed by gravity continuously through a tubular container and in counterflow to the gaseous acid. The container increases in cross section so as to accommodate the volumetric expansion of the uranium oxide during its transformation into the fluoride, thus preventing clogging of the apparatus, which had hitherto rendered impracticable commercial use of the reaction. The procedure and apparatus are described. To ensure satisfactory corrosion-resistance, nickel or 'Monel' (nickel 67 per cent., copper 30 per cent., iron and manganese about 1 per cent. each) is stipulated for all parts of the apparatus with which the reaction constituents or products come into contact.

COMMISSARIAT A L'ENERGIE ATOMIQUE
(inventors, P. VERTES, V. SAUTERON, M. DELANGE).
Brit. Pat. 800,692.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.

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